Electronic Supplementary Material (ESI) for RSC Advances.


# Supporting Information

## Synthesis of ionic liquid-supported hypervalent iodine reagent and its application as 'catch and release' reagent for α-substituted acetophenones

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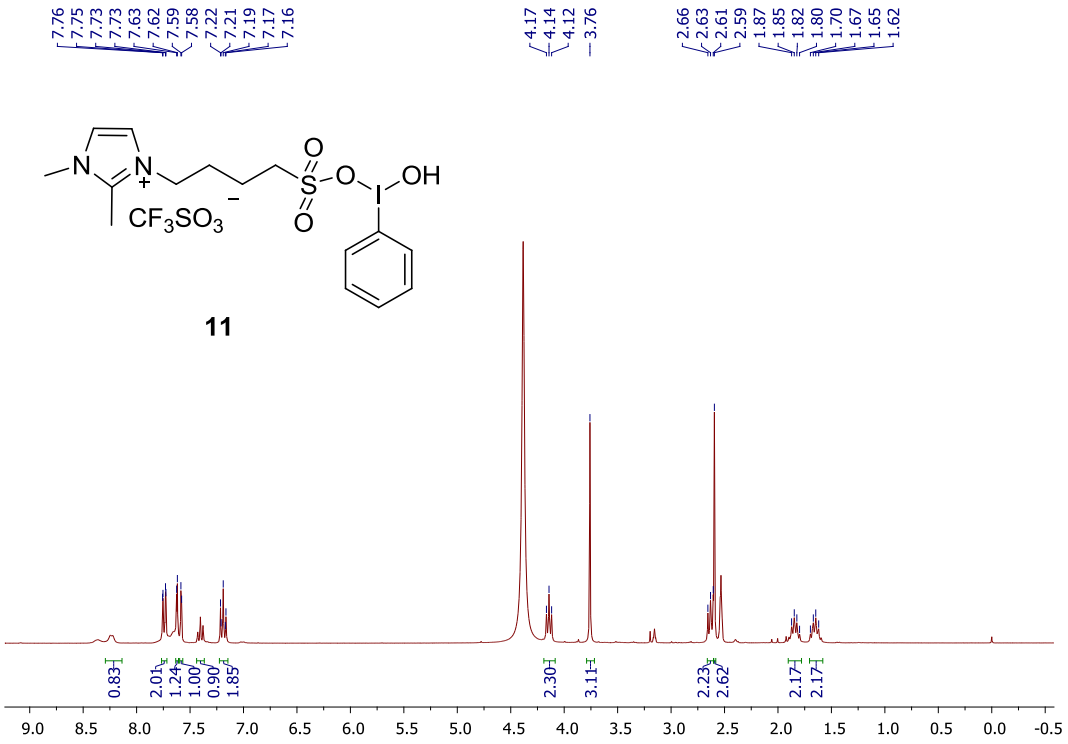
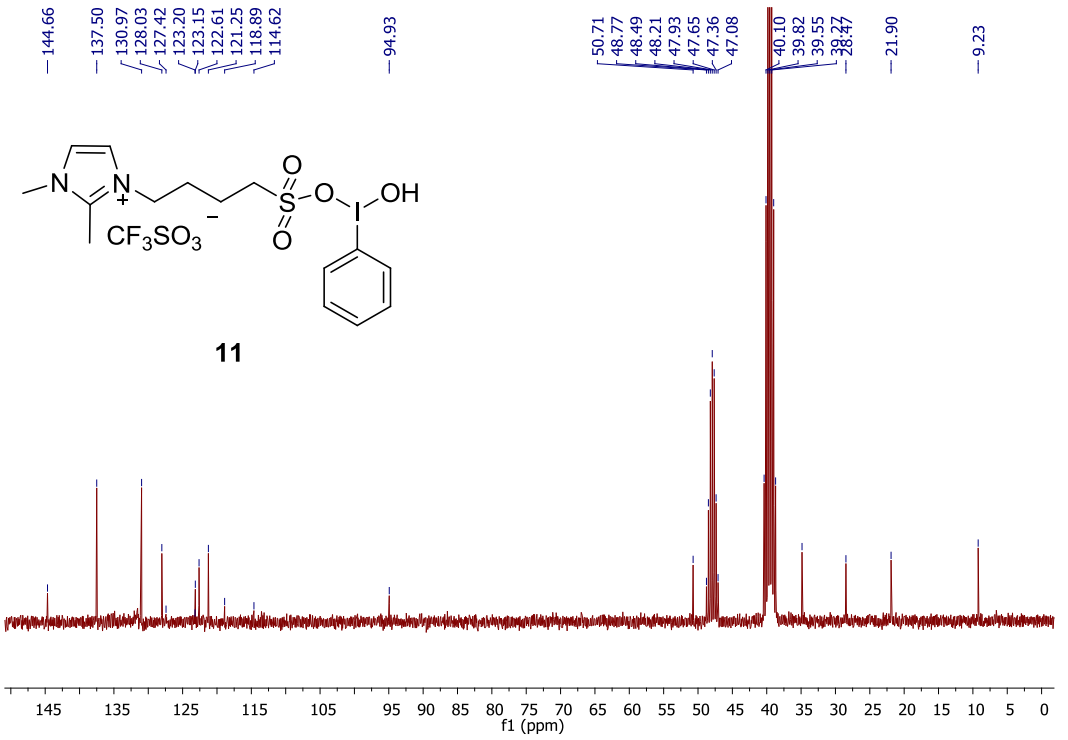
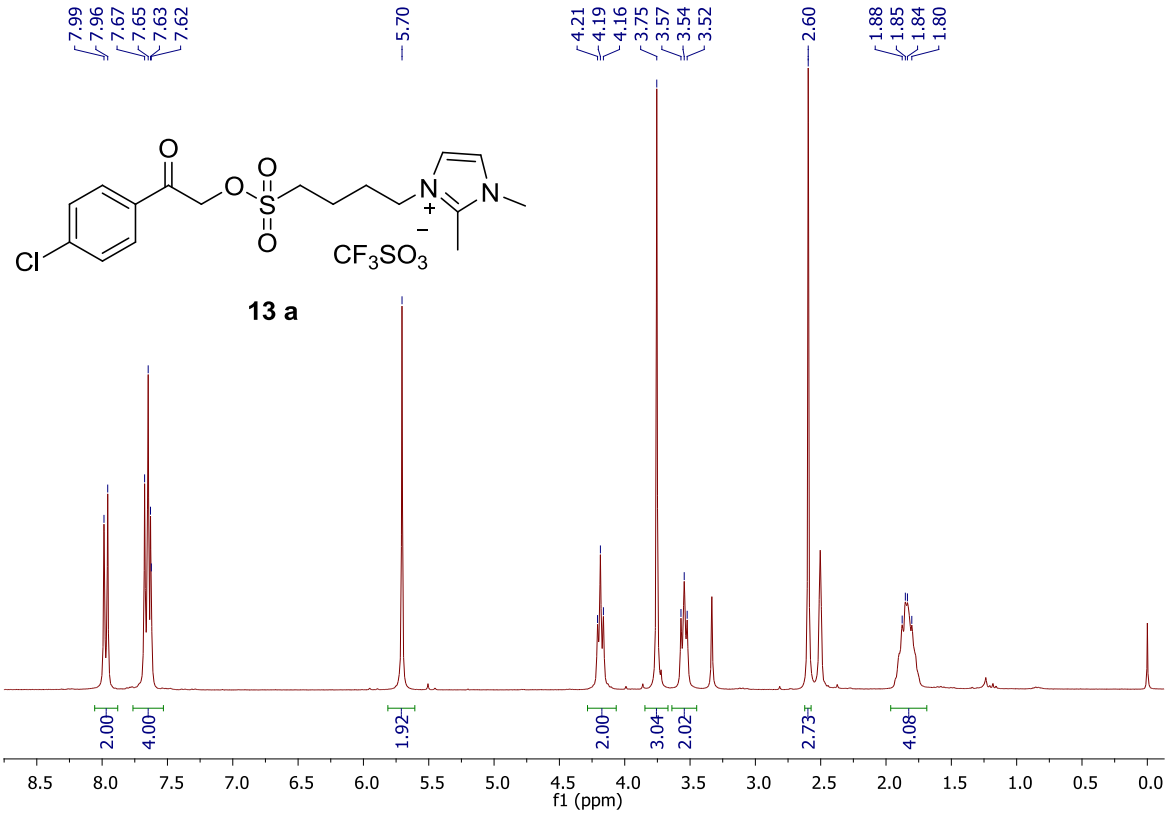
### 1. General informatin

The NMR spectra were recorded on 300 MHz, 400 MHz and 500 MHz spectrometers using $CDCl_3$, DMSO-$d_6$ and $CD_3OD$ as solvents. The chemical shifts were expressed in ppm. Reactions were monitored by thin-layer chromatography (TLC) carried out on silica-coated aluminum plates (60F-254) using UV light as visualizing agent. High resolution mass spectra (HRMS) were recorded on a mass spectrometer using electrospray ionization-time of flight (ESI-TOF). Melting points were determined on open capillary tube on automated melting point apparatus and are uncorrected. All the chemicals and reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated.

## 2. Copies of $^{1}H$ and $^{13}C$ NMR spectra

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1.85
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O
O
S
O
O
N
+
N
N
Cl
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4.00
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2.00
3.04
2.02
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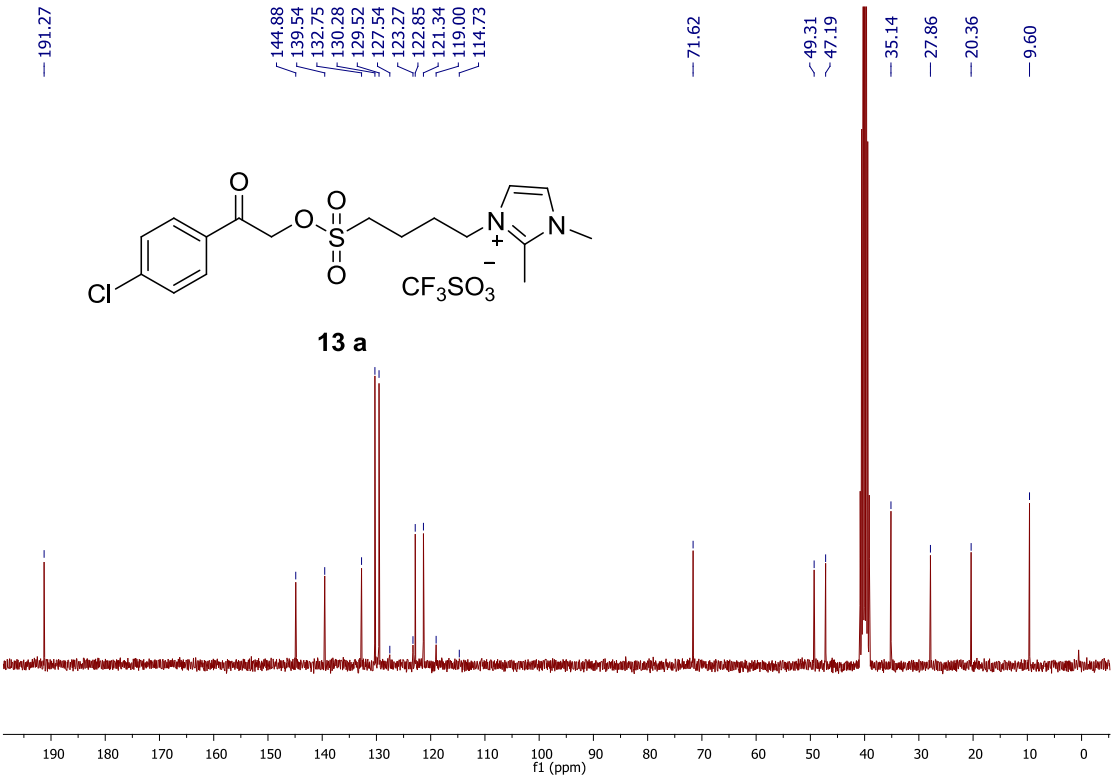
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47.19
35.14
27.86
20.36
9.60
O
O
O
S
O
O
N
+
N
N
Cl
CF3SO3
13 a
190
180
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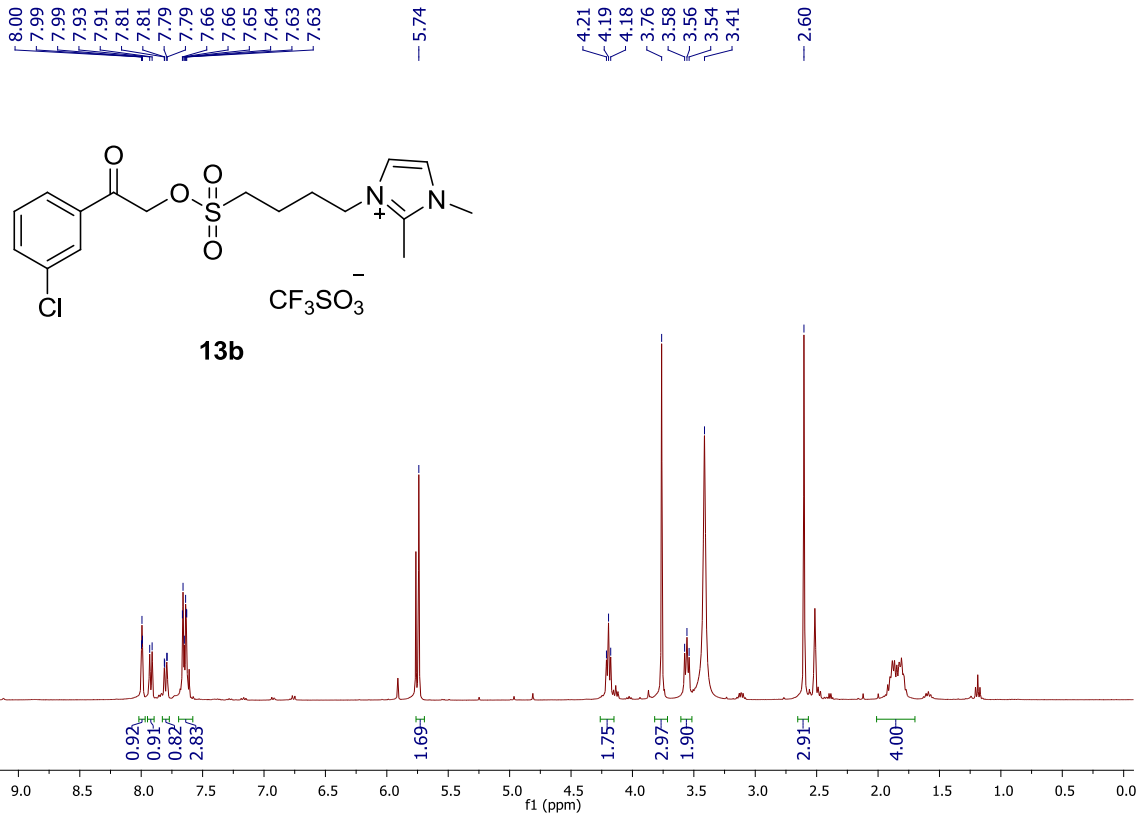
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5.74
4.21
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3.58
3.56
3.54
3.41
2.60
O
O
O
S
O
O
N
+
N
Cl
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−
13b
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2.91
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7.5
7.0
6.5
6.0
5.5
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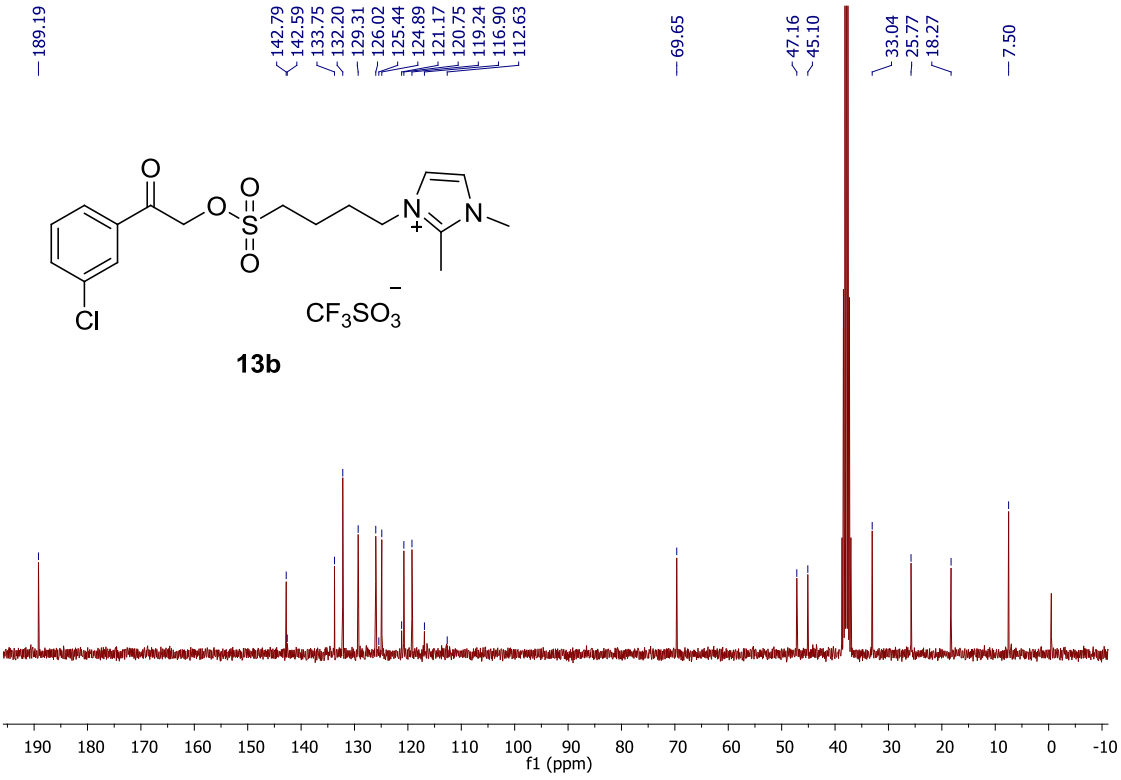
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O
O
O
S
O
O
N
+
N
Cl
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−
13b
190
180
170
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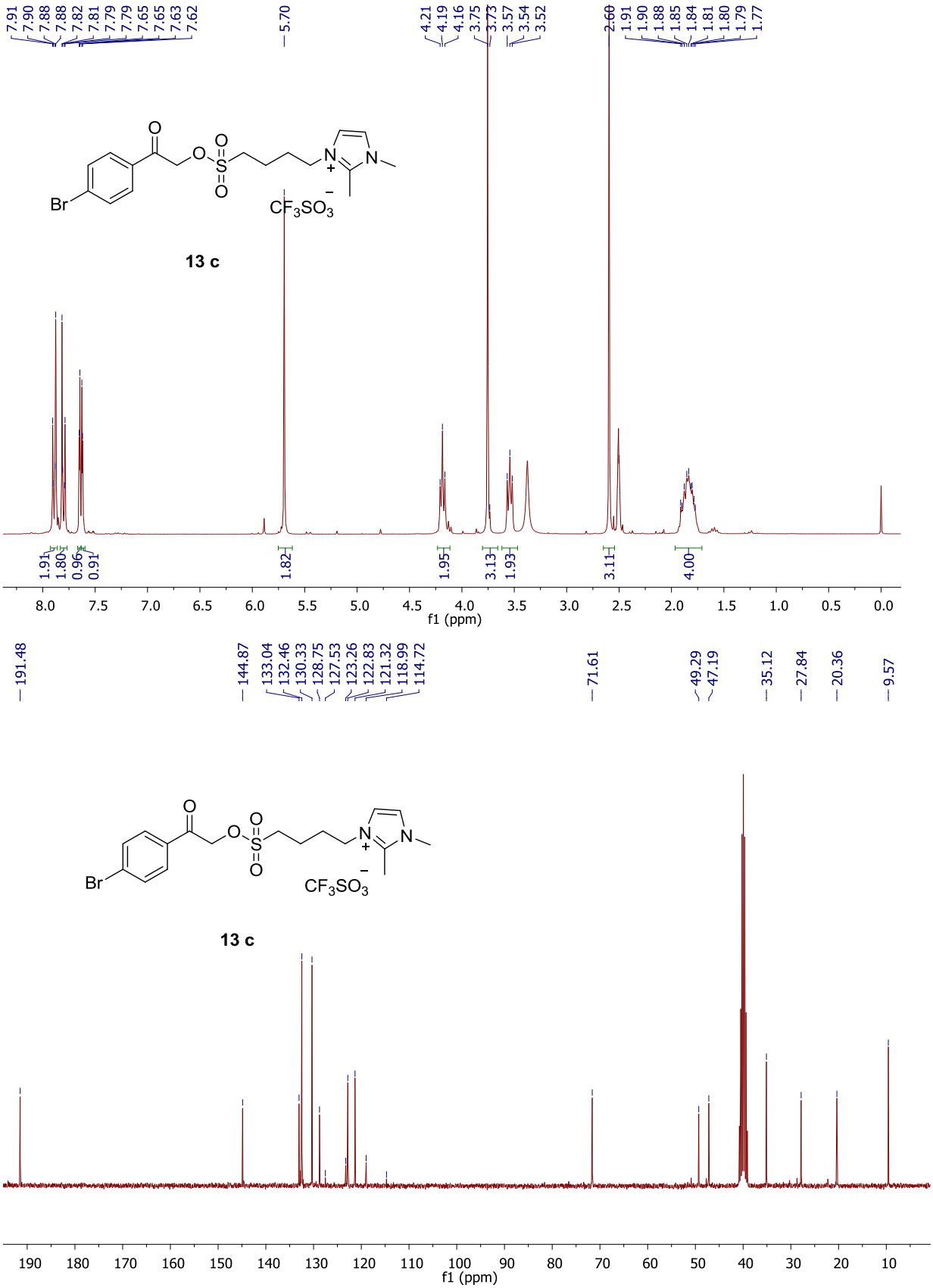
f1 (ppm)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3.13
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f1 (ppm)
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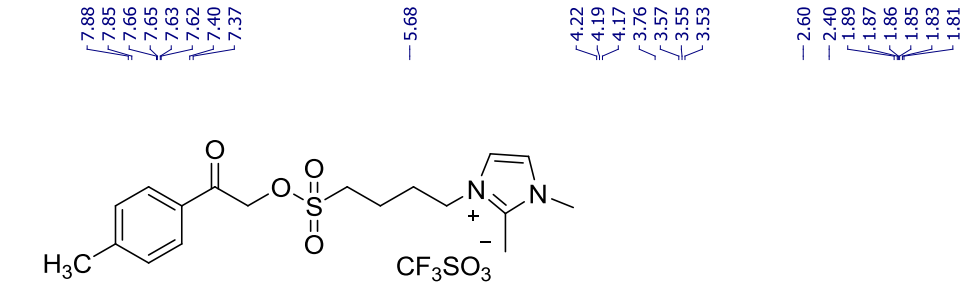
f1 (ppm)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1.87
1.86
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O
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N
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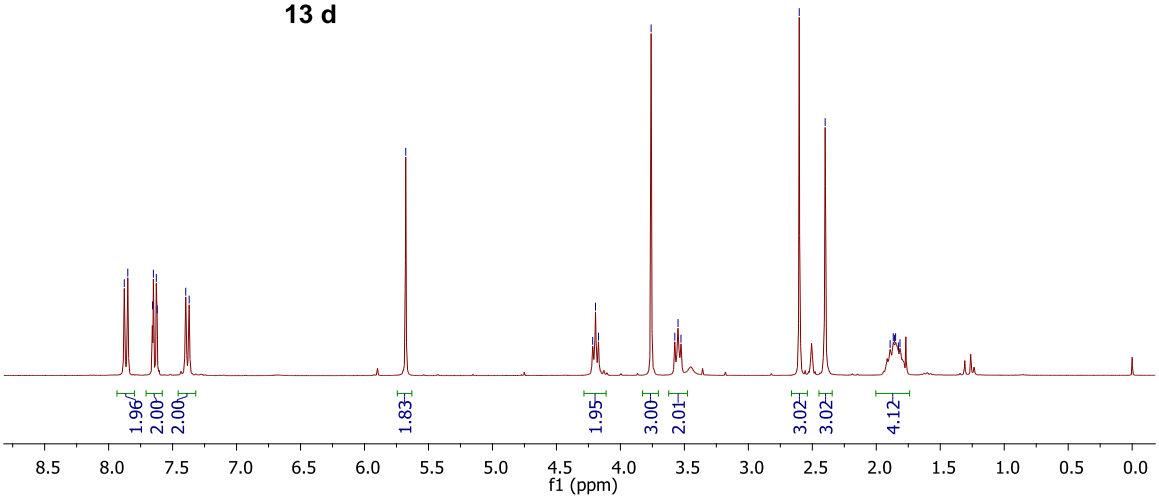
+
−
H3C
CF3SO3

**13 d**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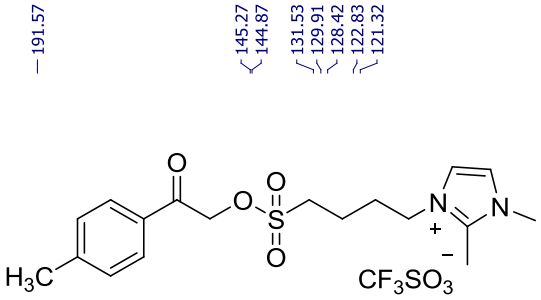
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O
O
O
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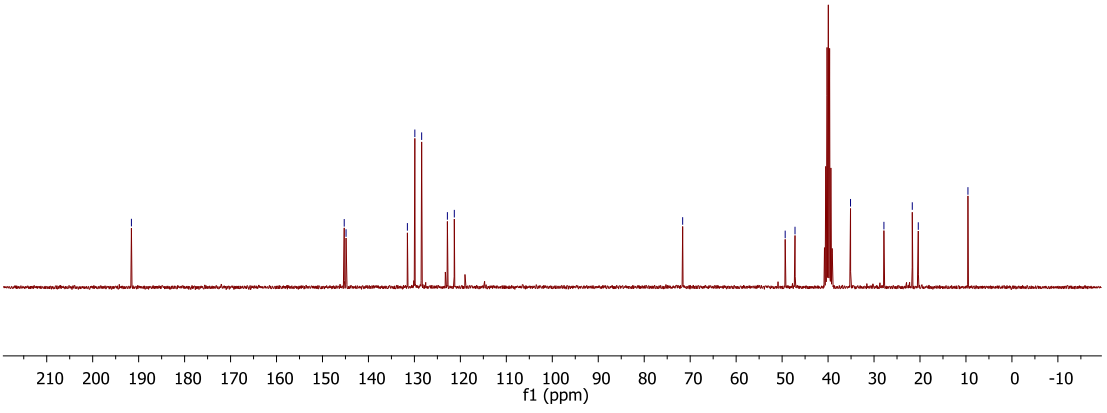
O
N
N
+
−
H3C
CF3SO3

**13 d**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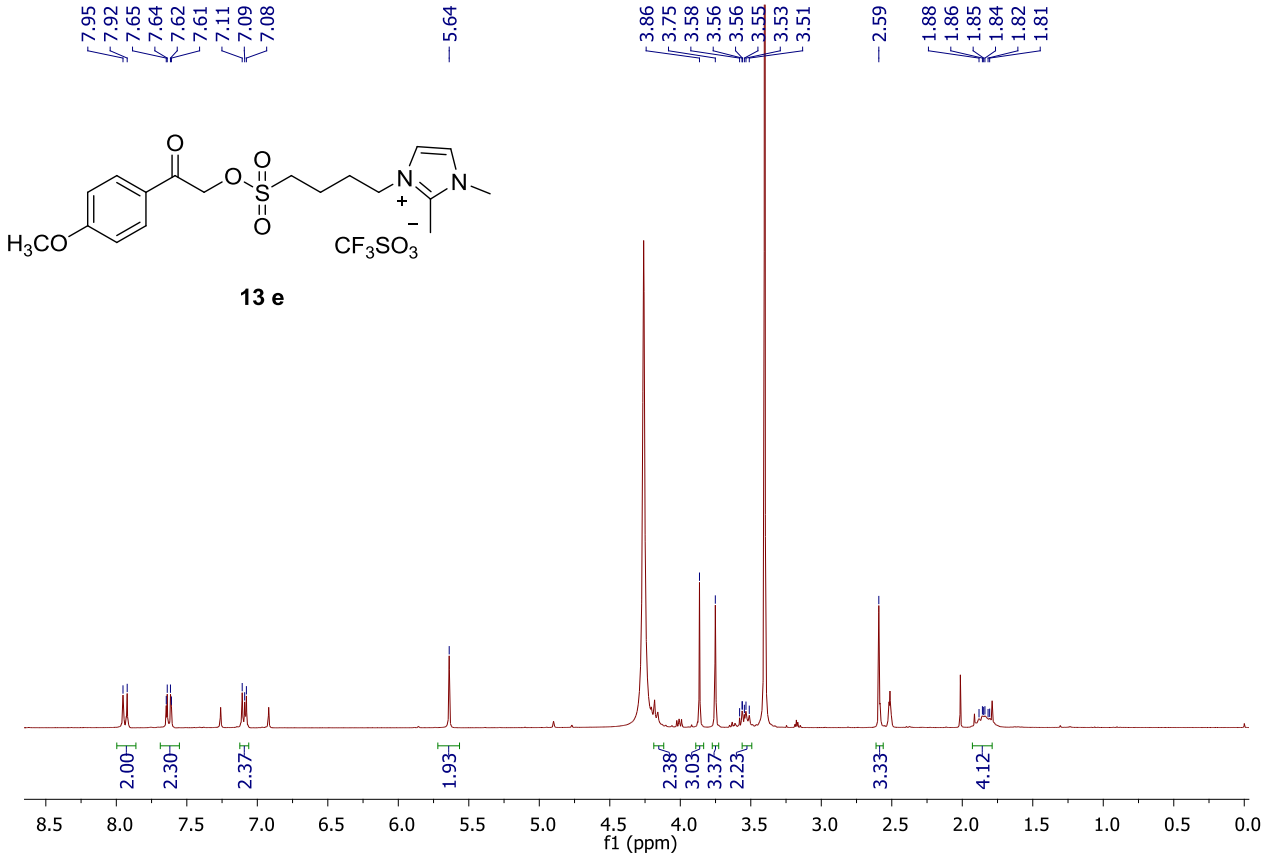
f1 (ppm)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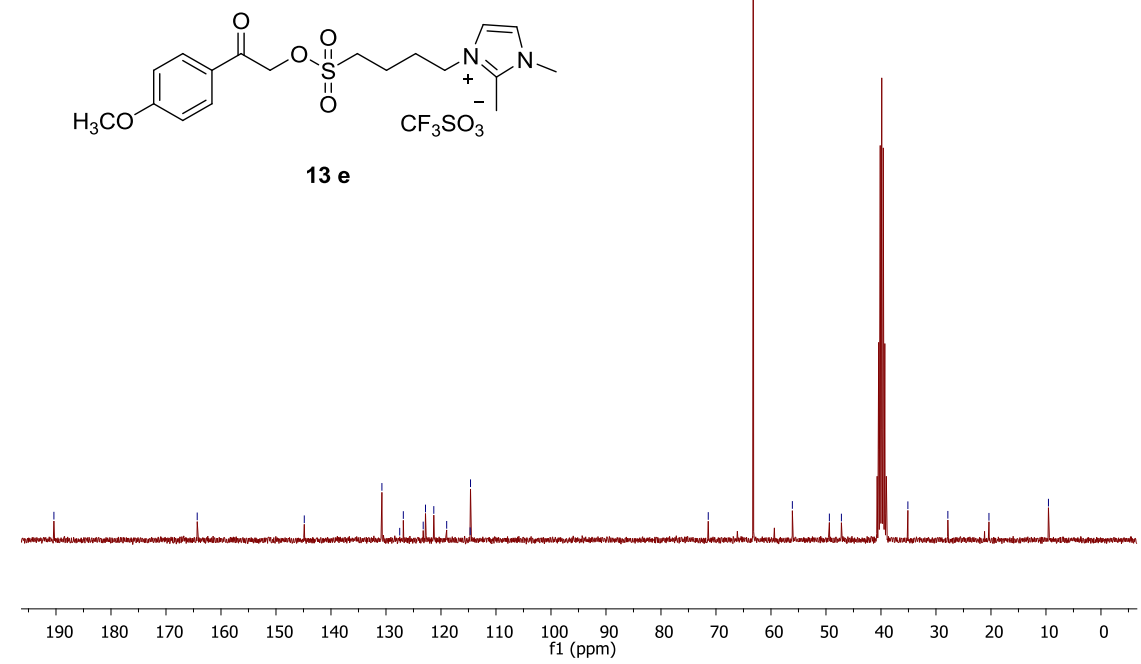
f1 (ppm)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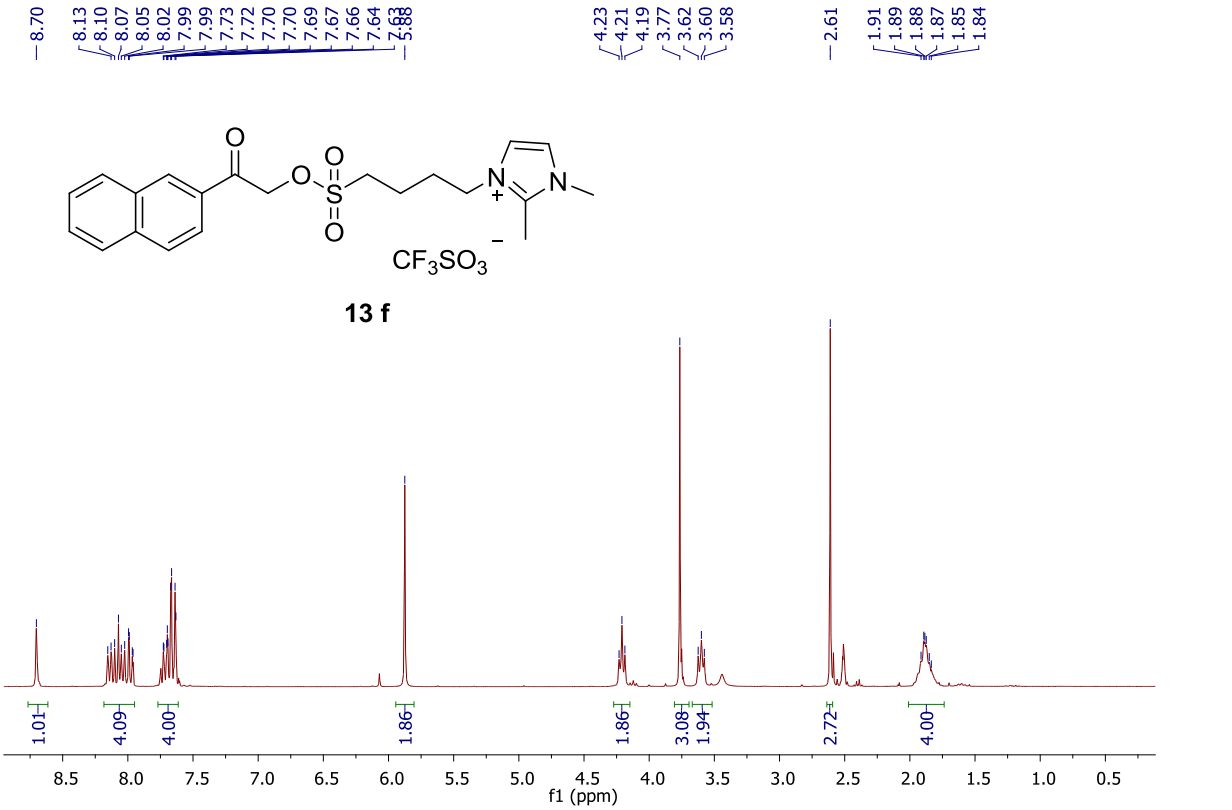
f1 (ppm)

**13 e**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1.84
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-
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4.09
4.00
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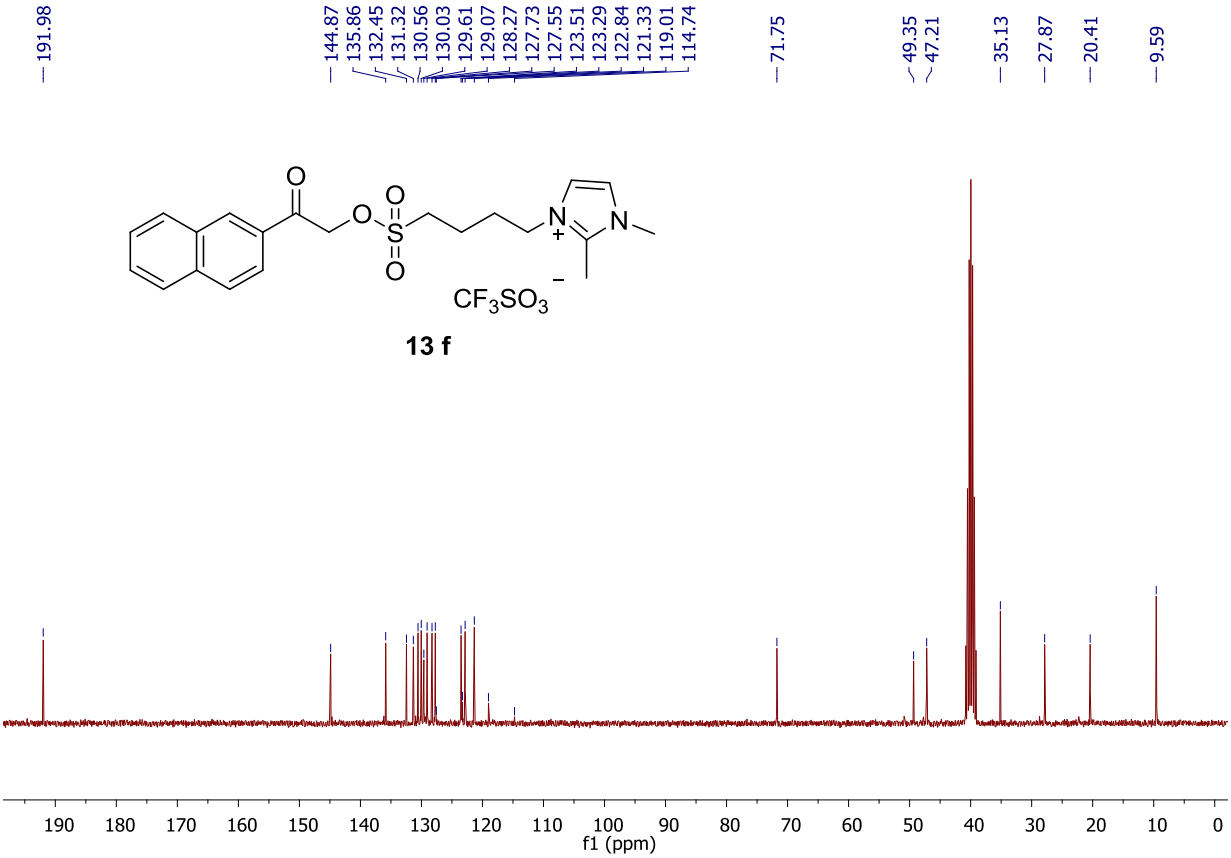
f1 (ppm)

**13 f**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O
O
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N
+
N
CF3SO3
-
190
180
170
160
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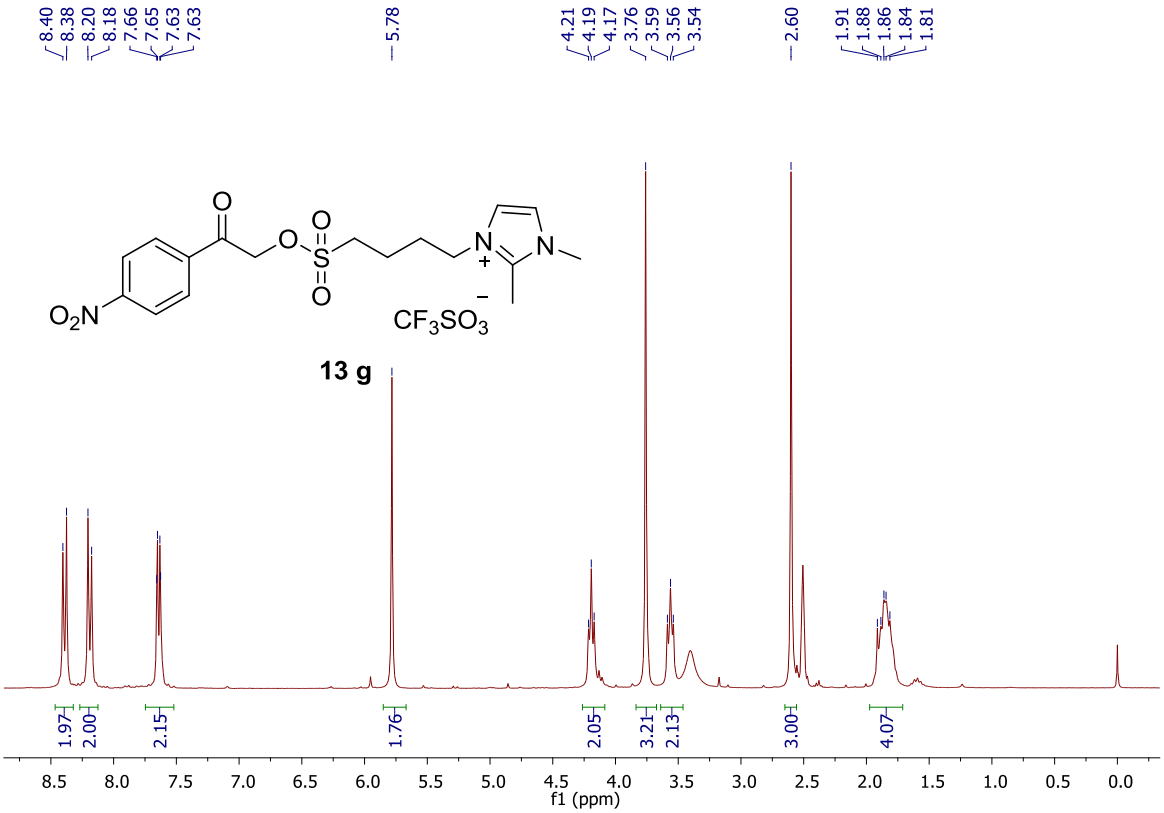
50
40
30
20
10
0
f1 (ppm)

**13 f**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3.56
3.54
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1.91
1.88
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1.84
1.81
O2N
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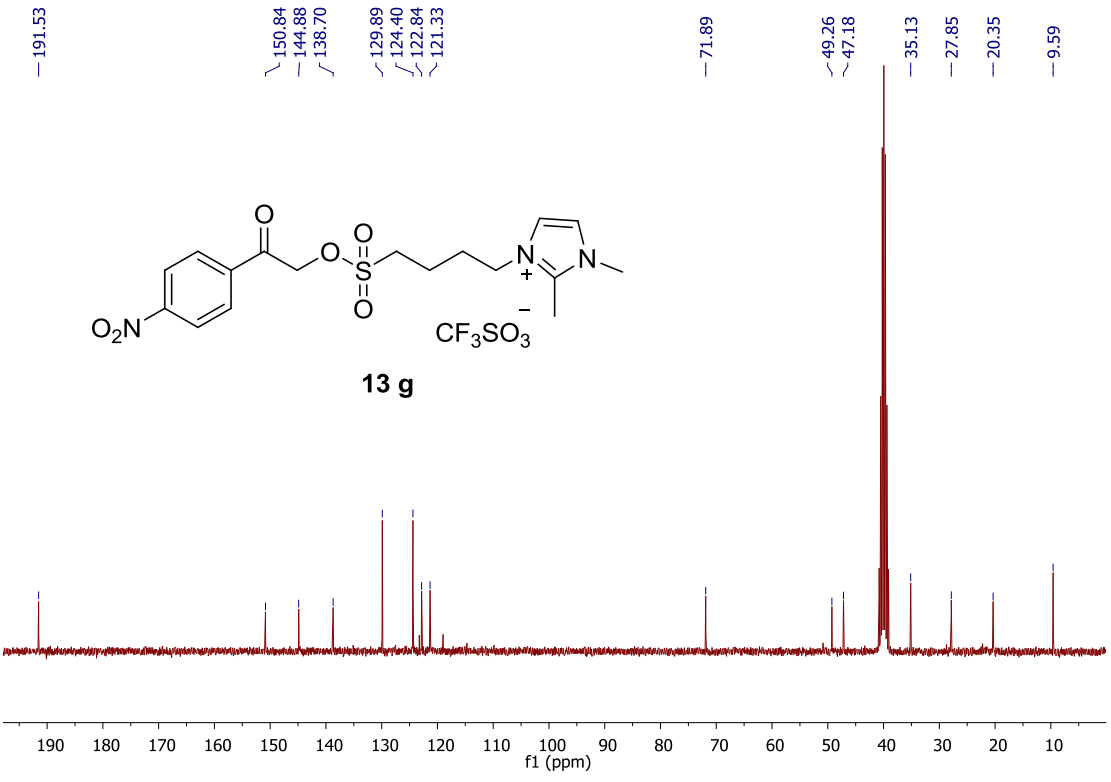
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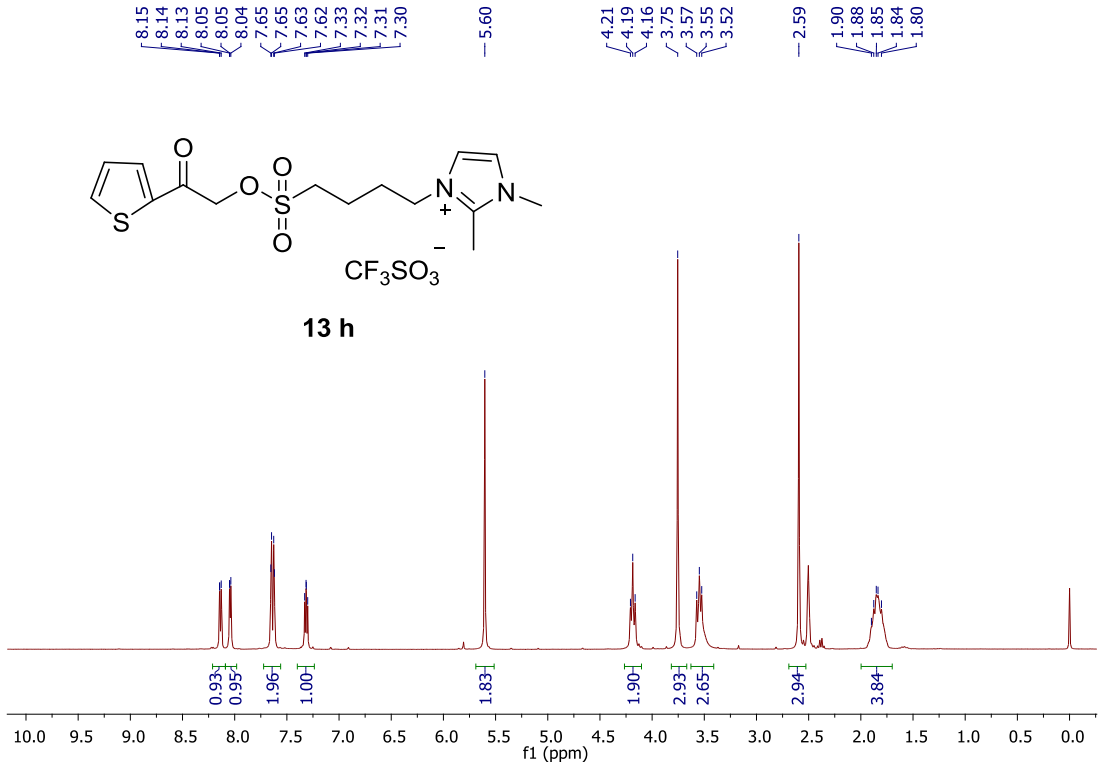
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N
+
−
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0.95
1.96
1.00
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2.93
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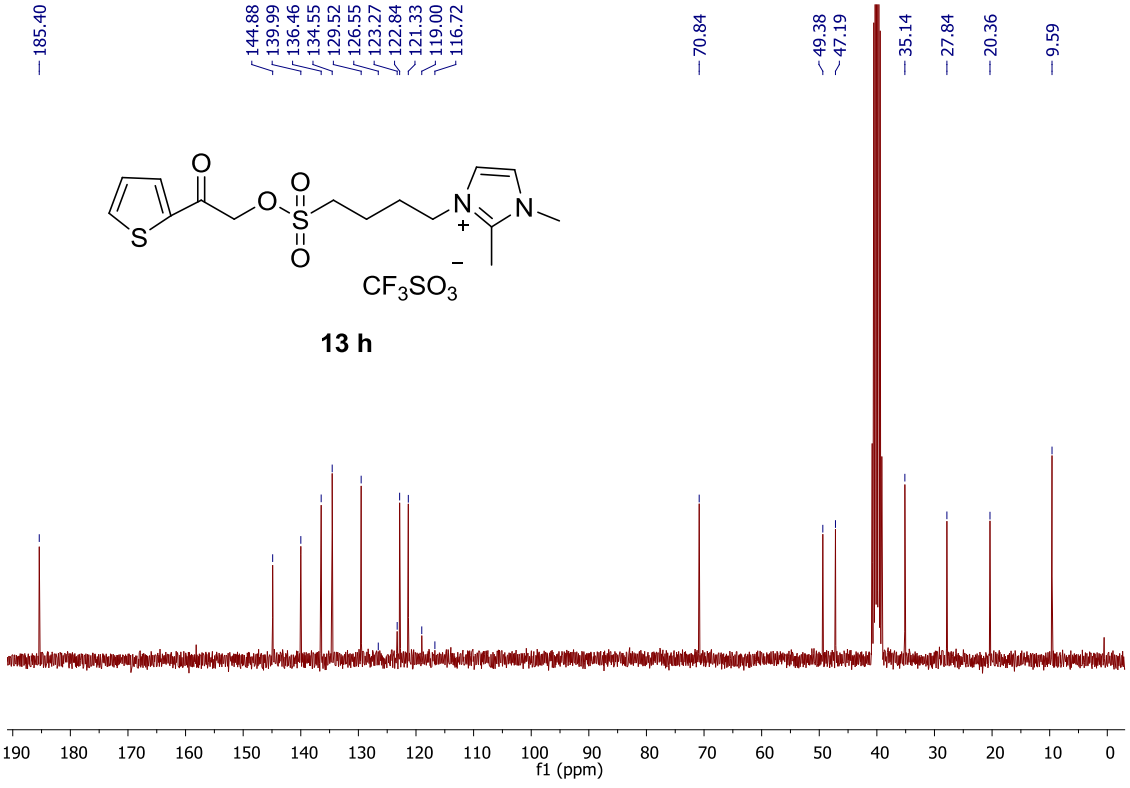
3.84
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

**13 h**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9.59
O
O
O
O
S
S
N
N
+
−
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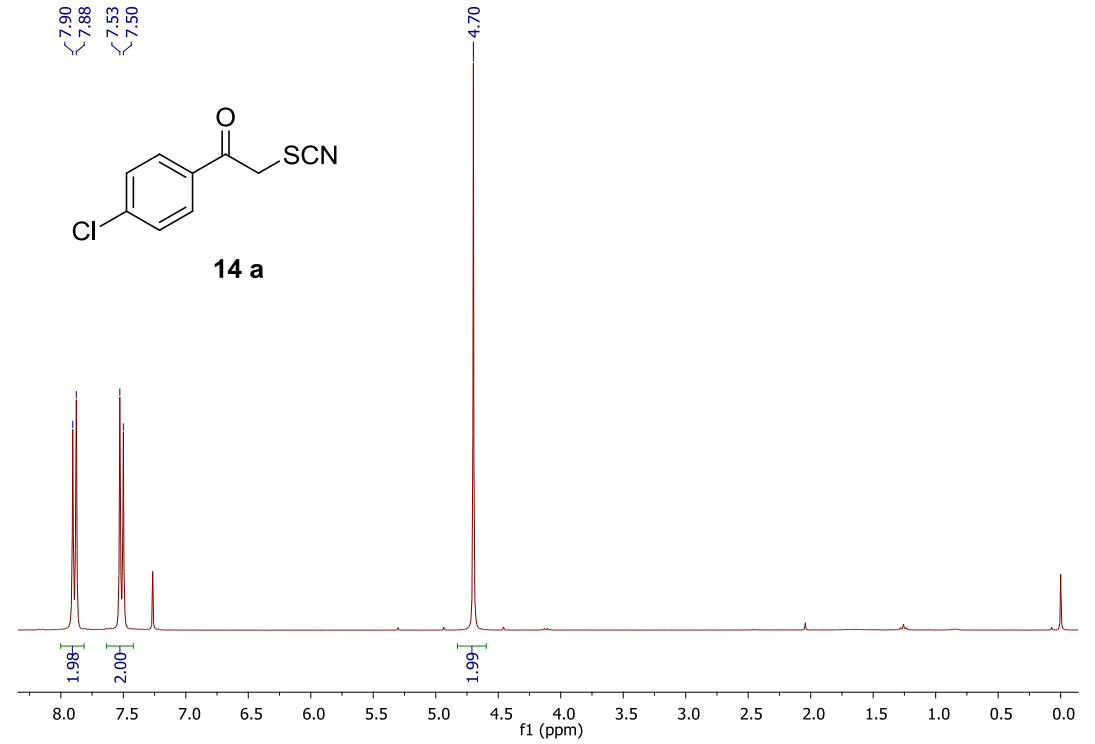
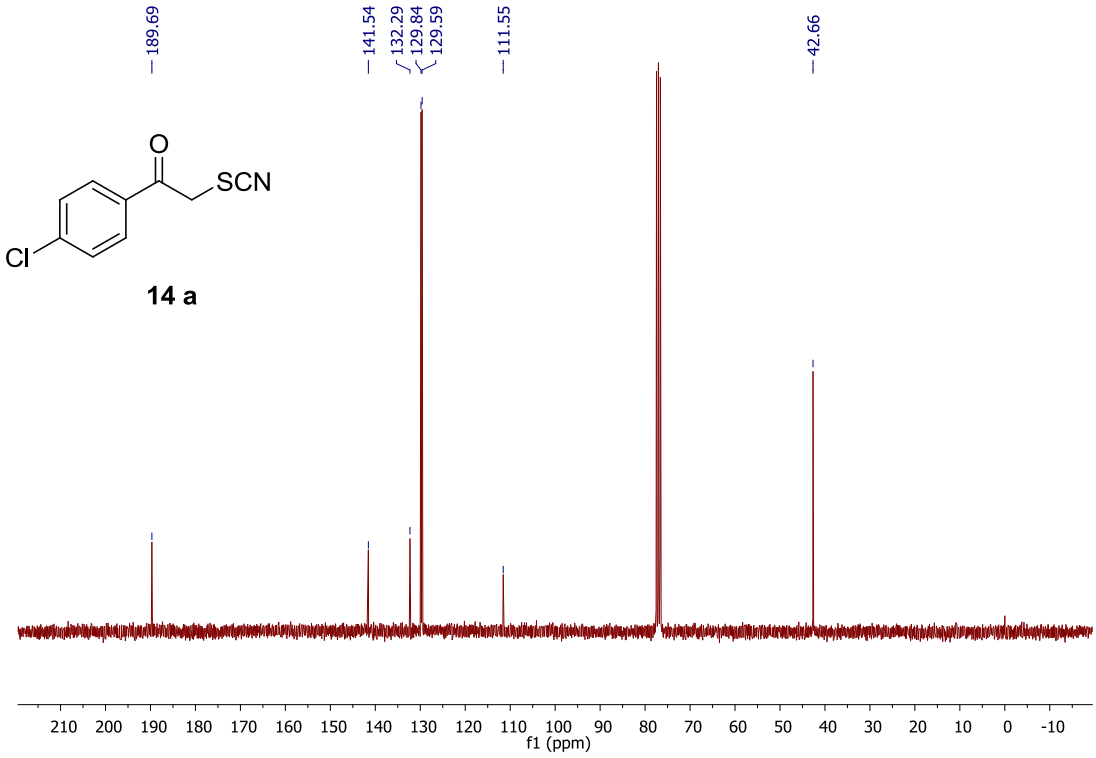
f1 (ppm)

**13 h**

1-(4-Chlorophenyl)-2-thiocyanatoethanone



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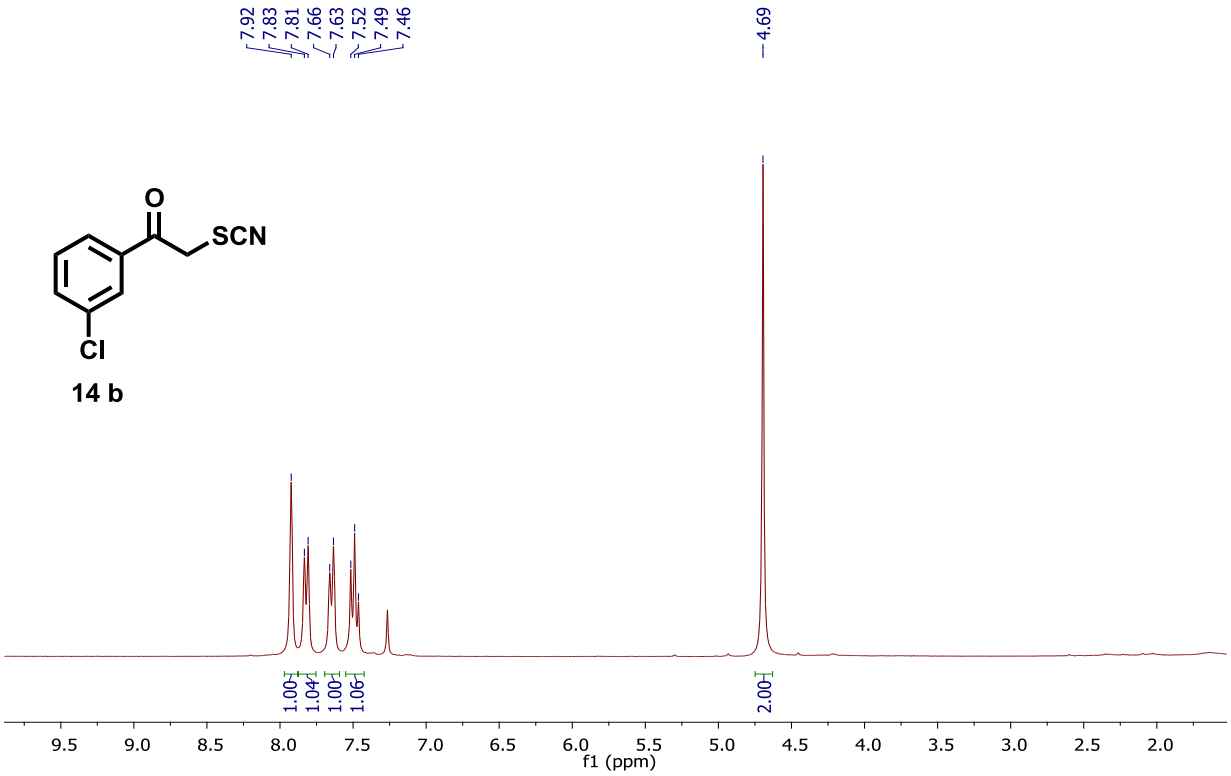


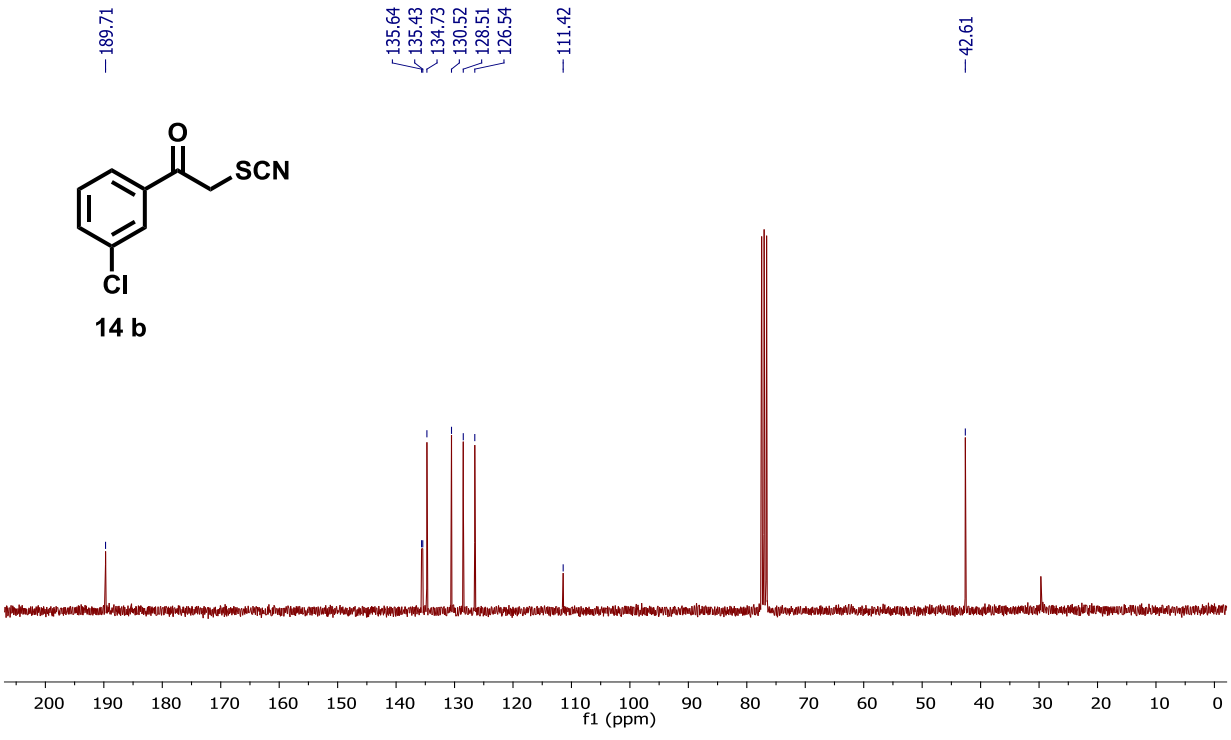

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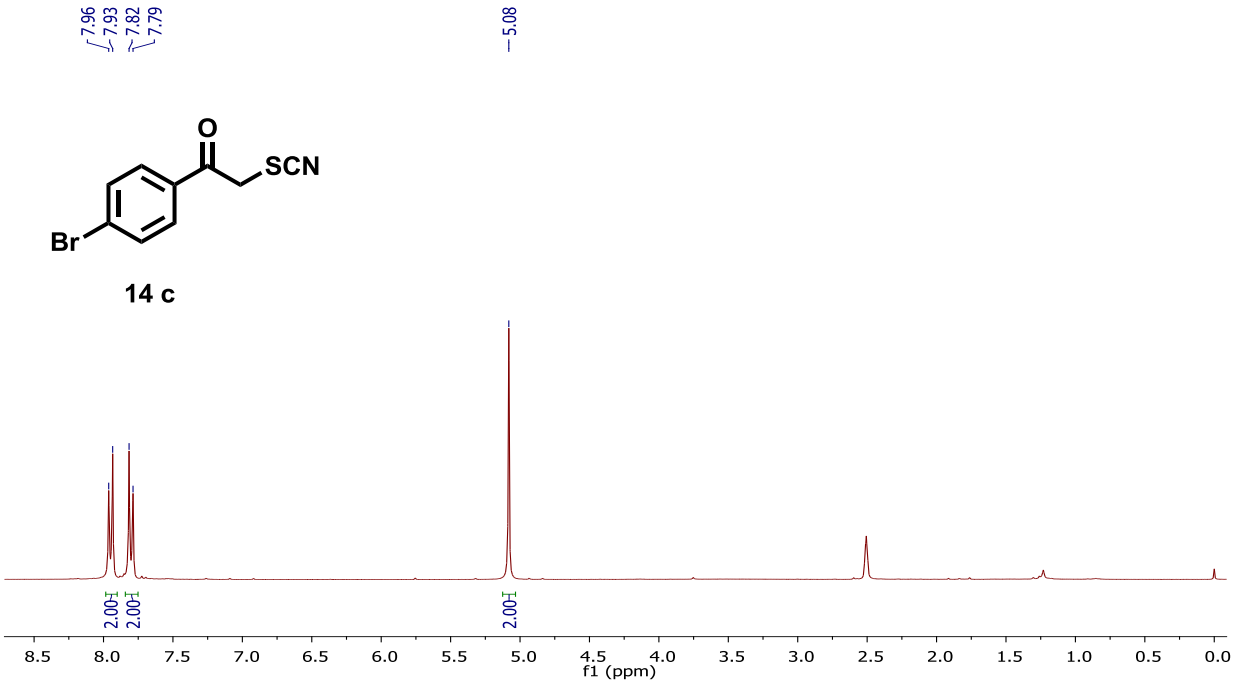
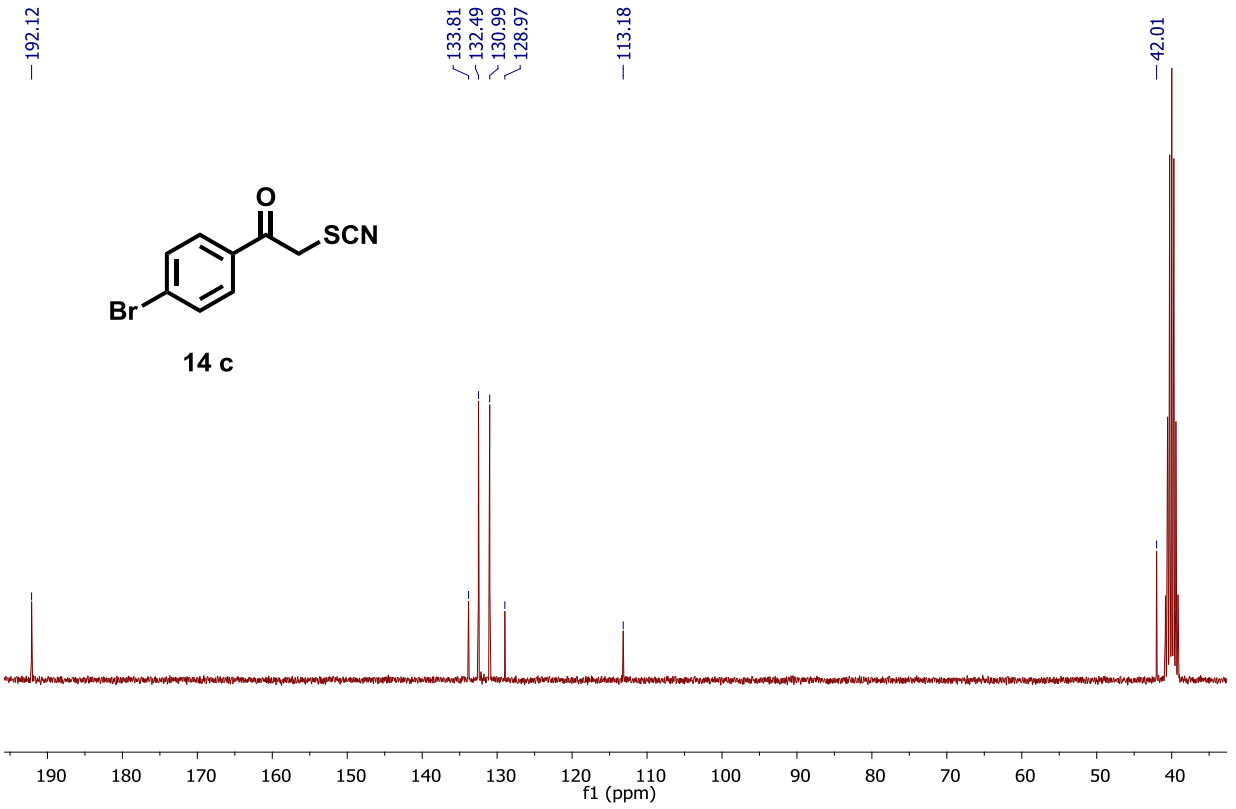
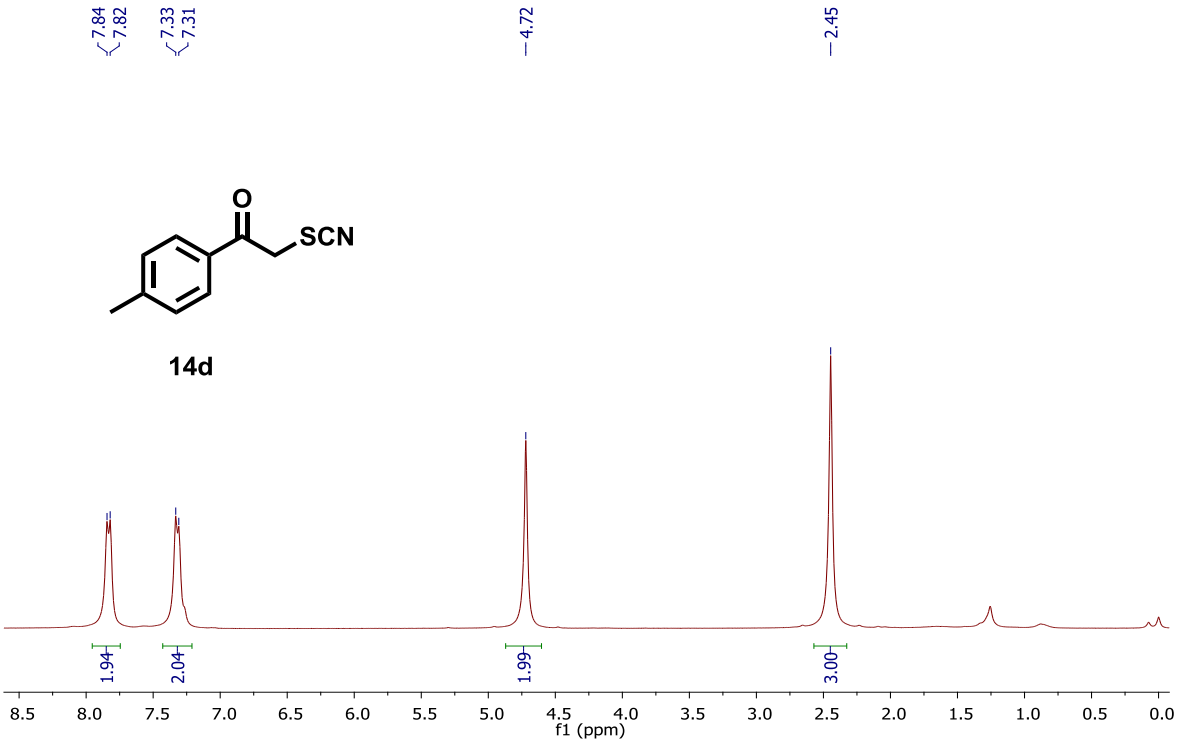
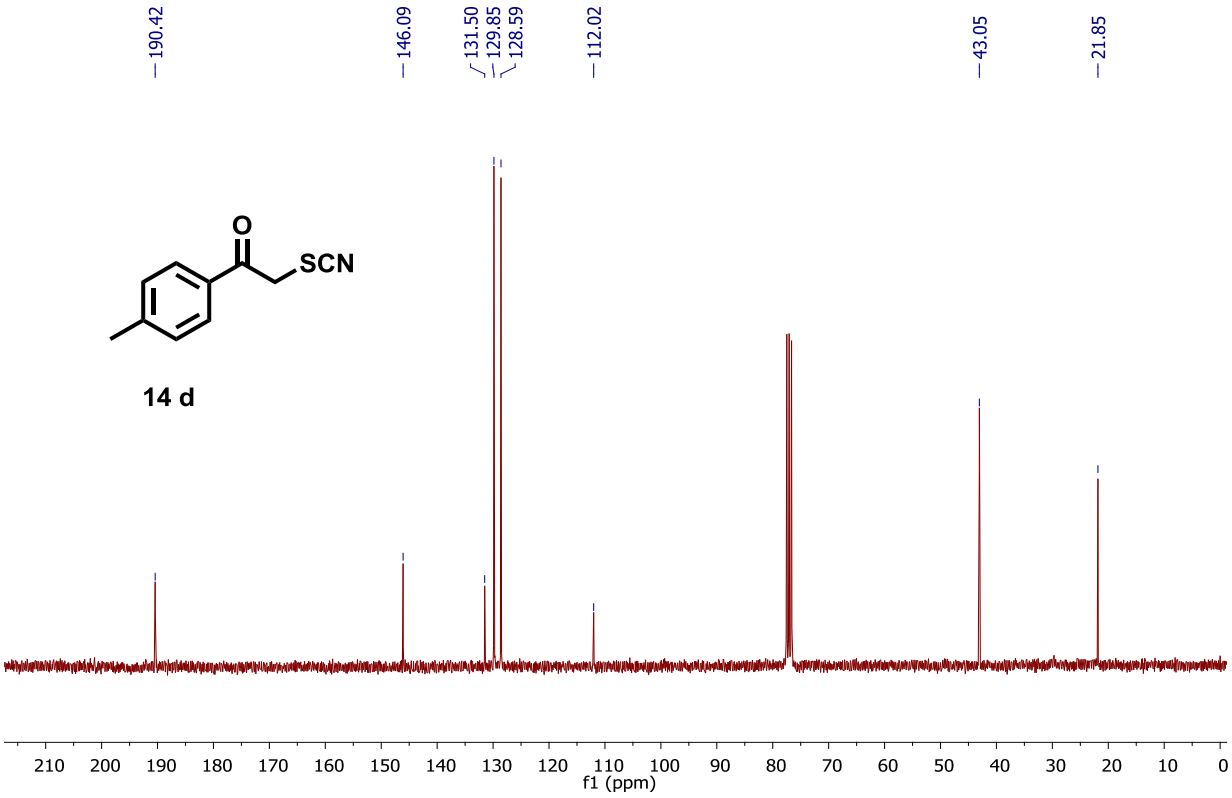


2-Thiocyanato-1-tolylethanone



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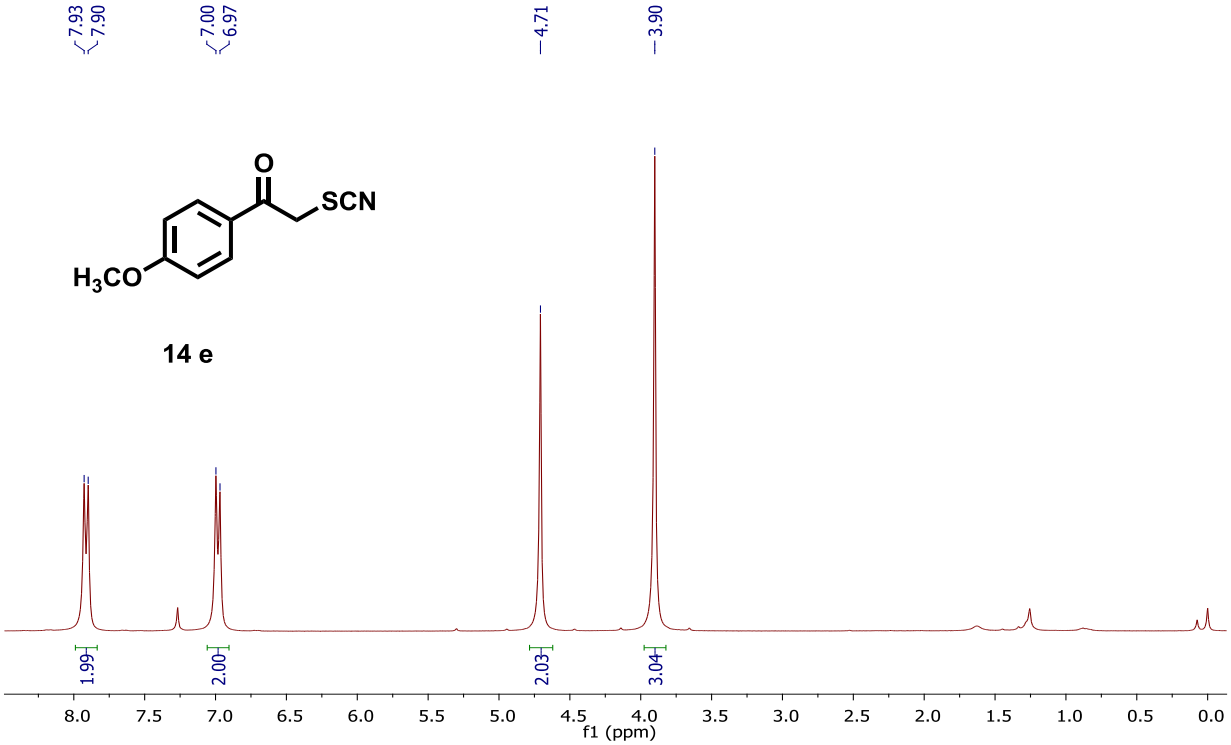


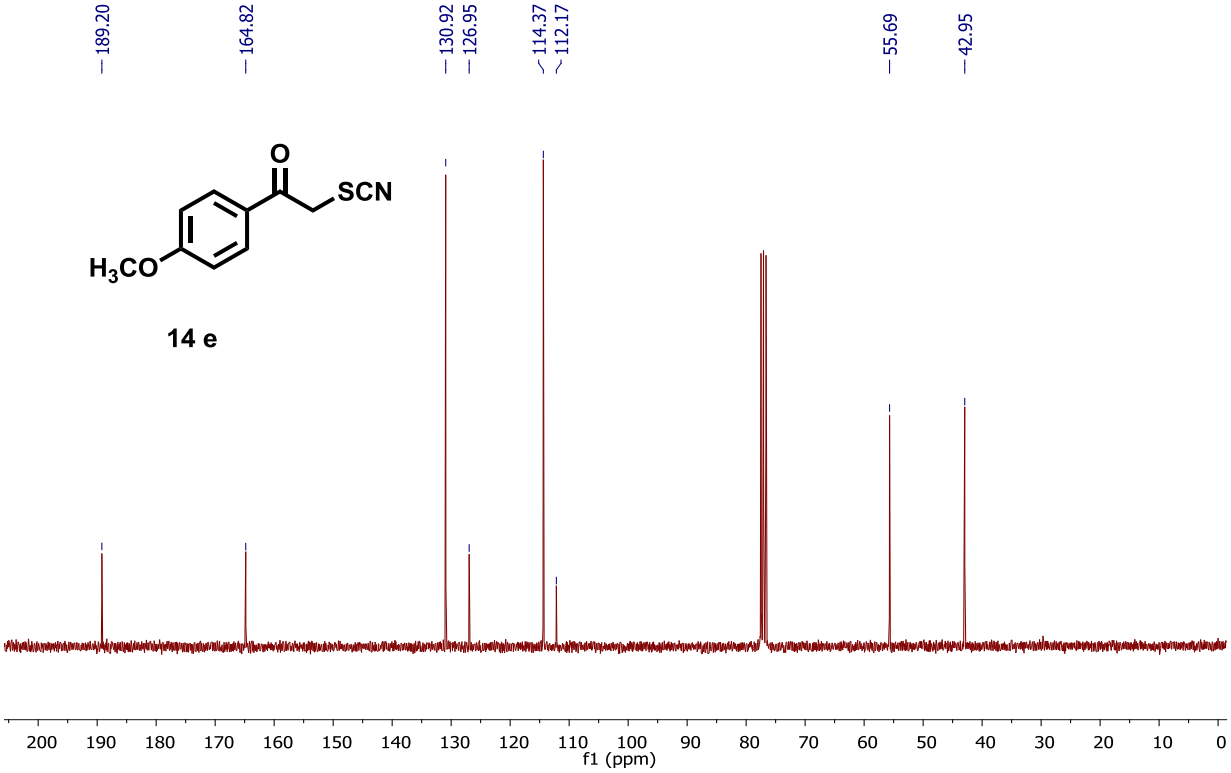

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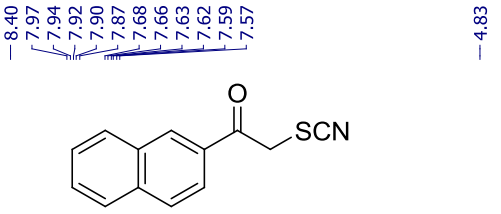
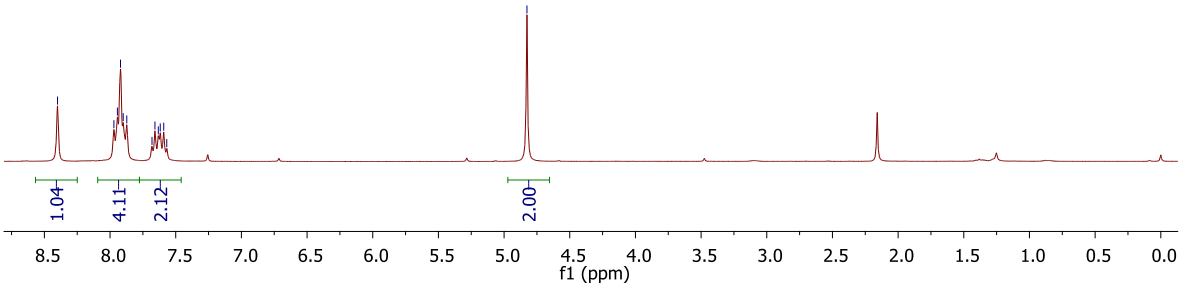
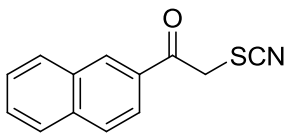


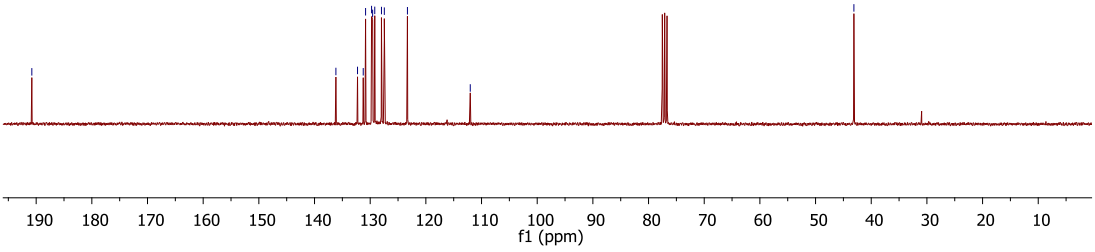

**14 f**



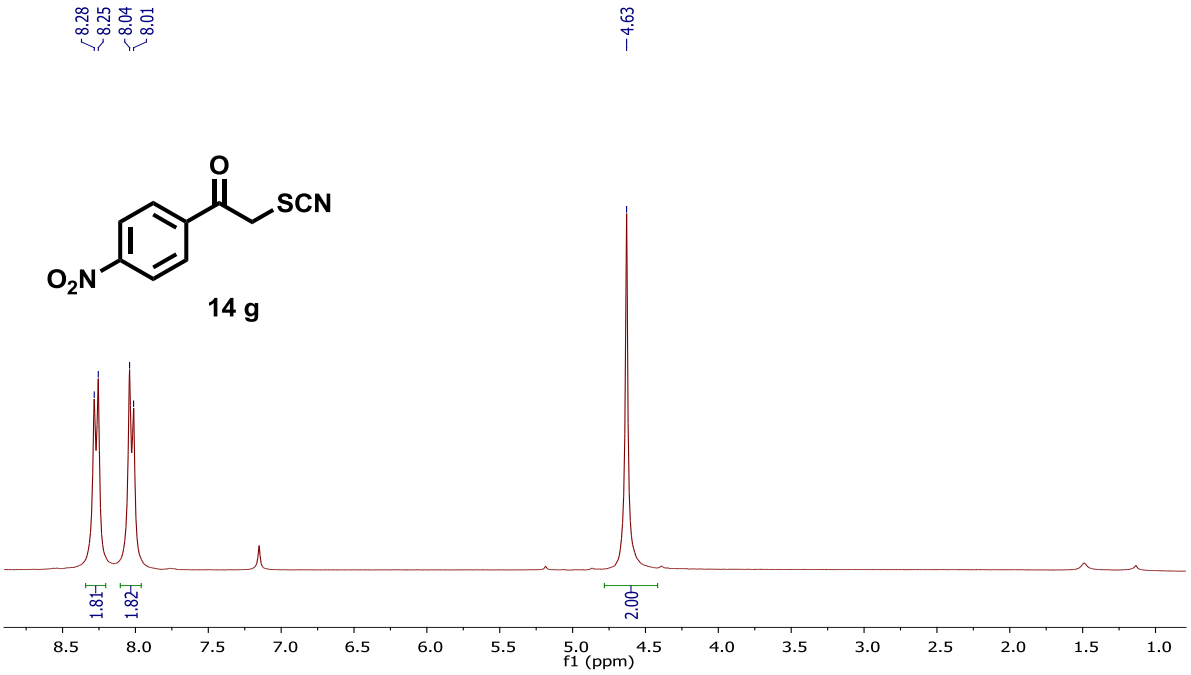


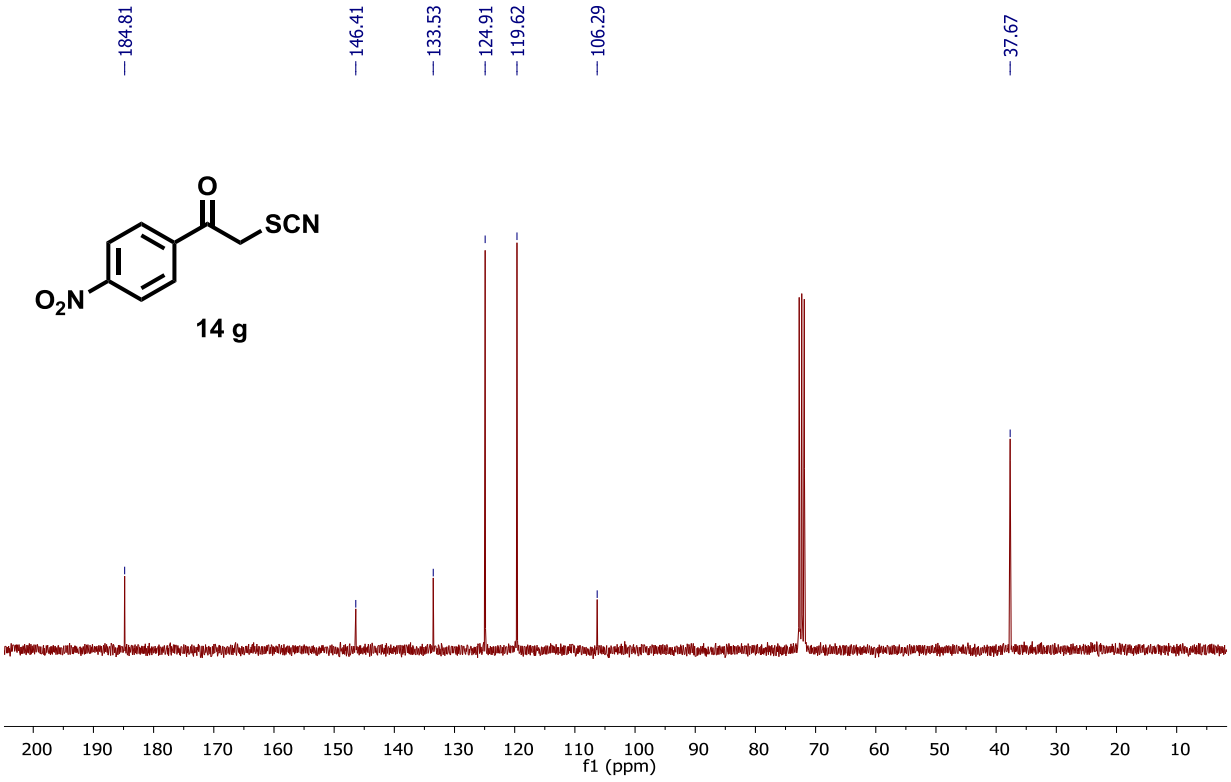



**14 f**

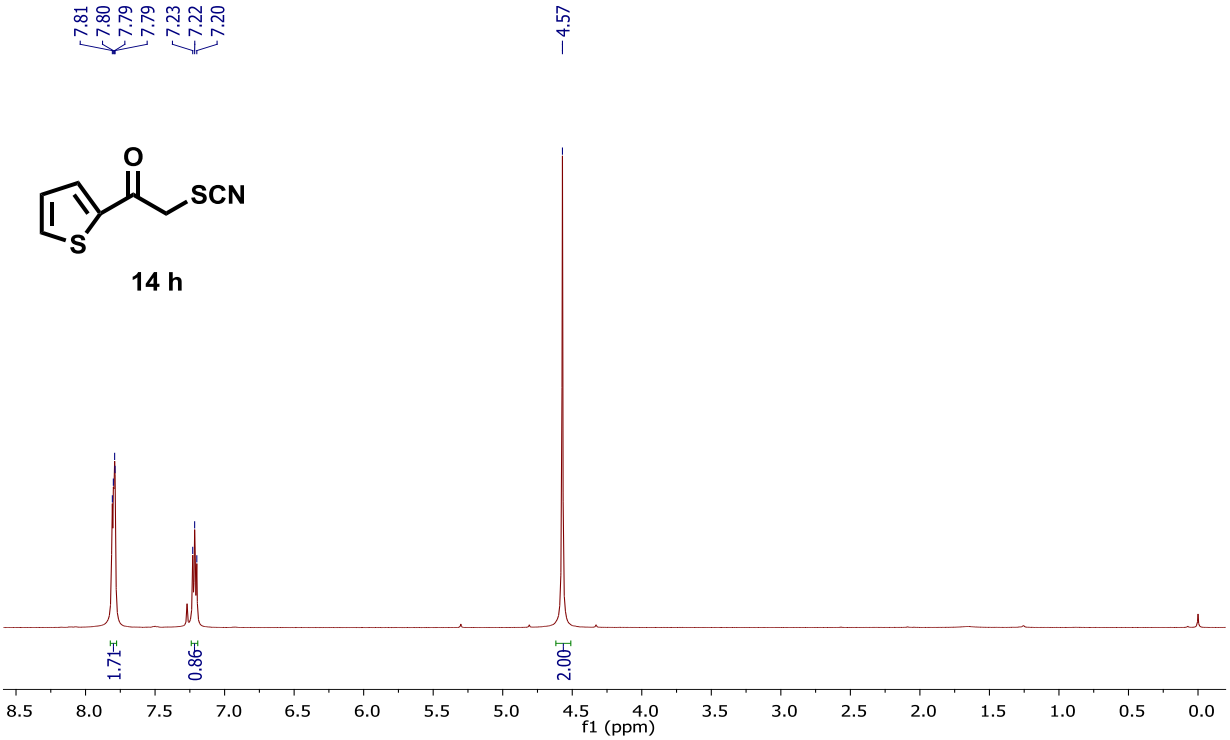


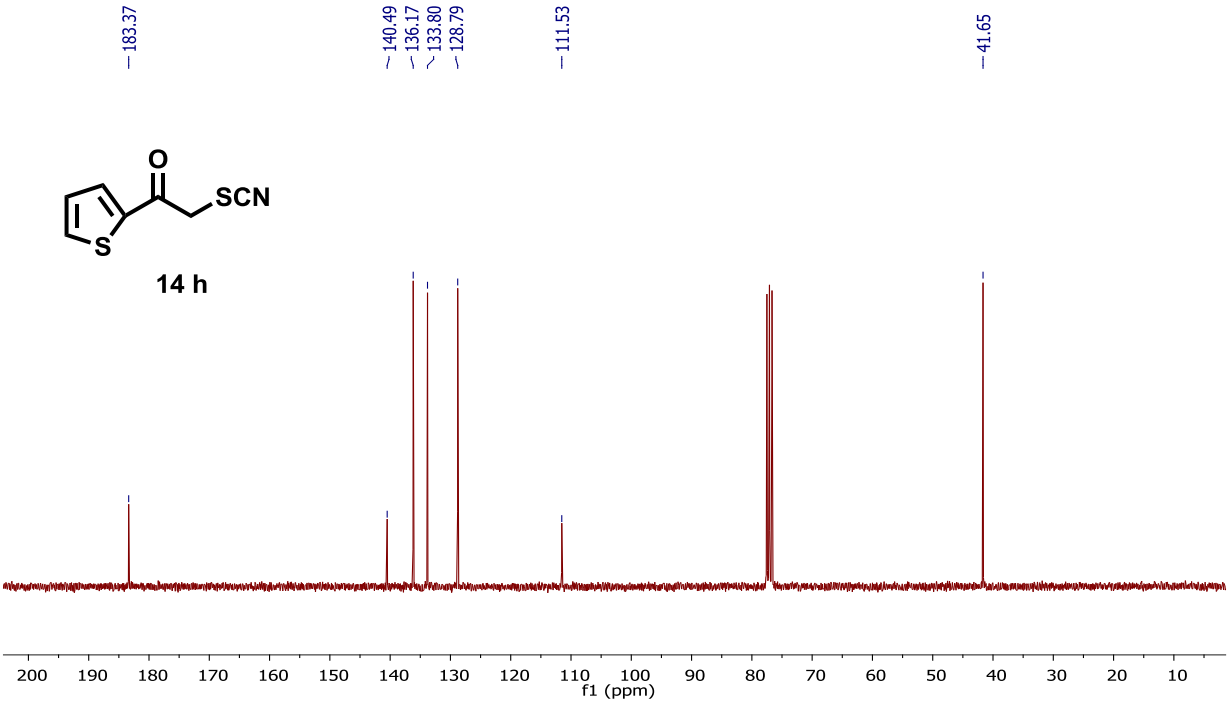
1-(4-Nitrophenyl)-2-thiocyanatoethanone



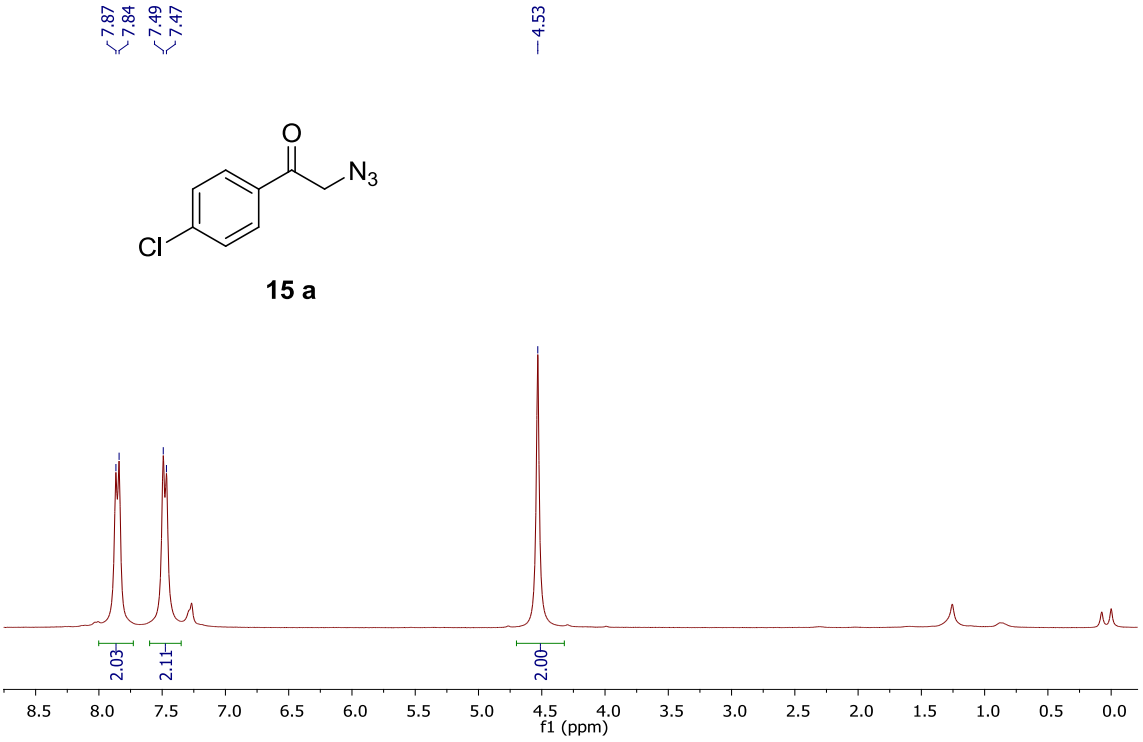



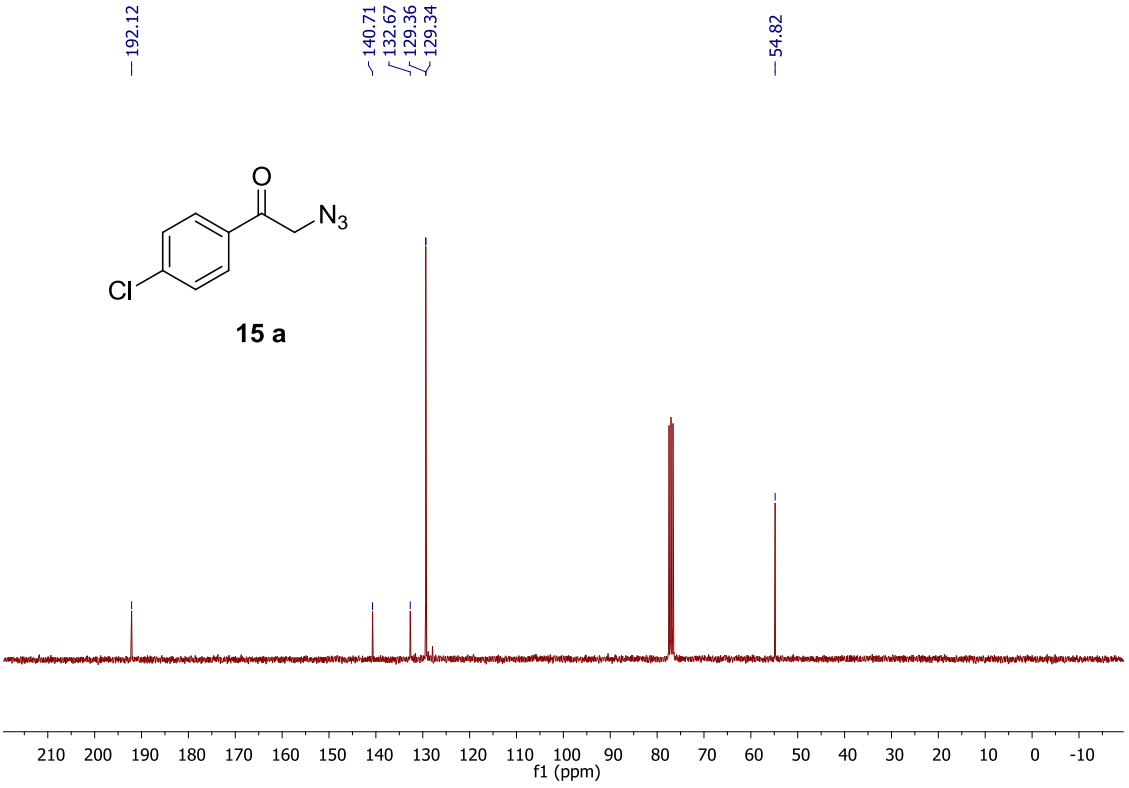
2-Thiocyanato-1-(thiophen-2-yl)ethanone



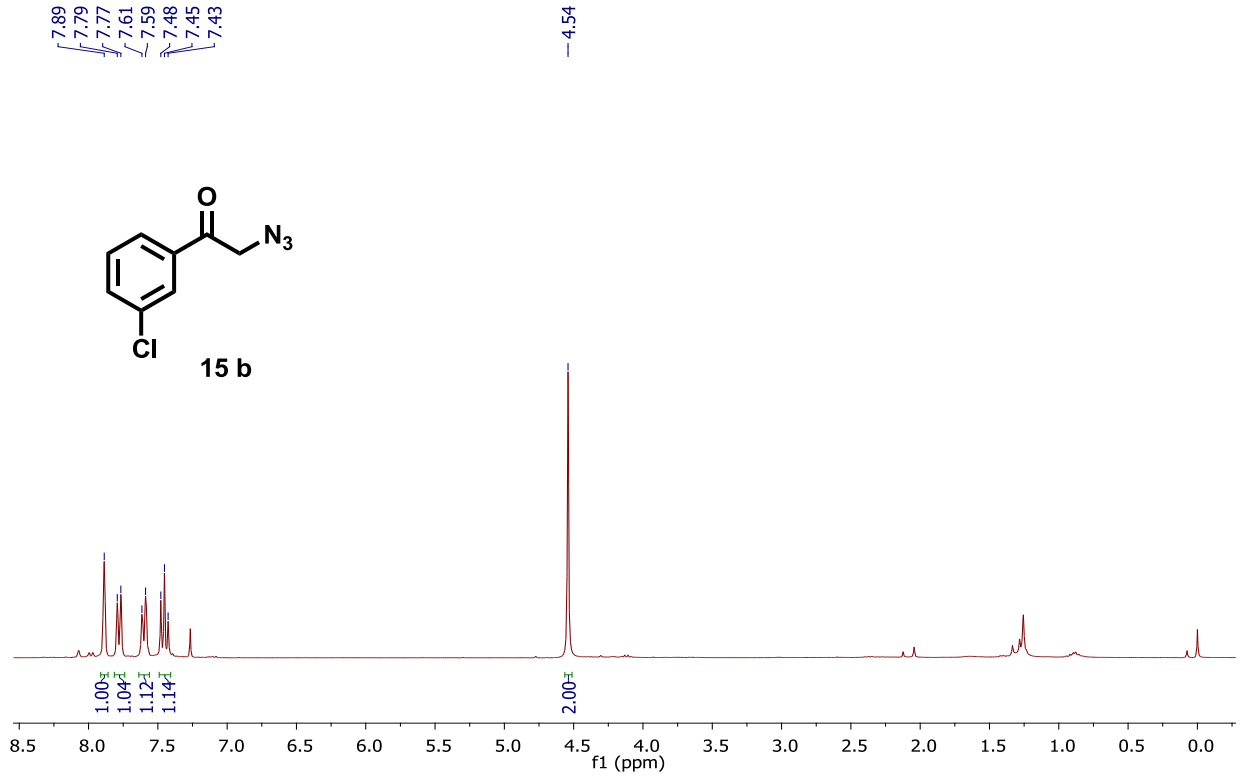



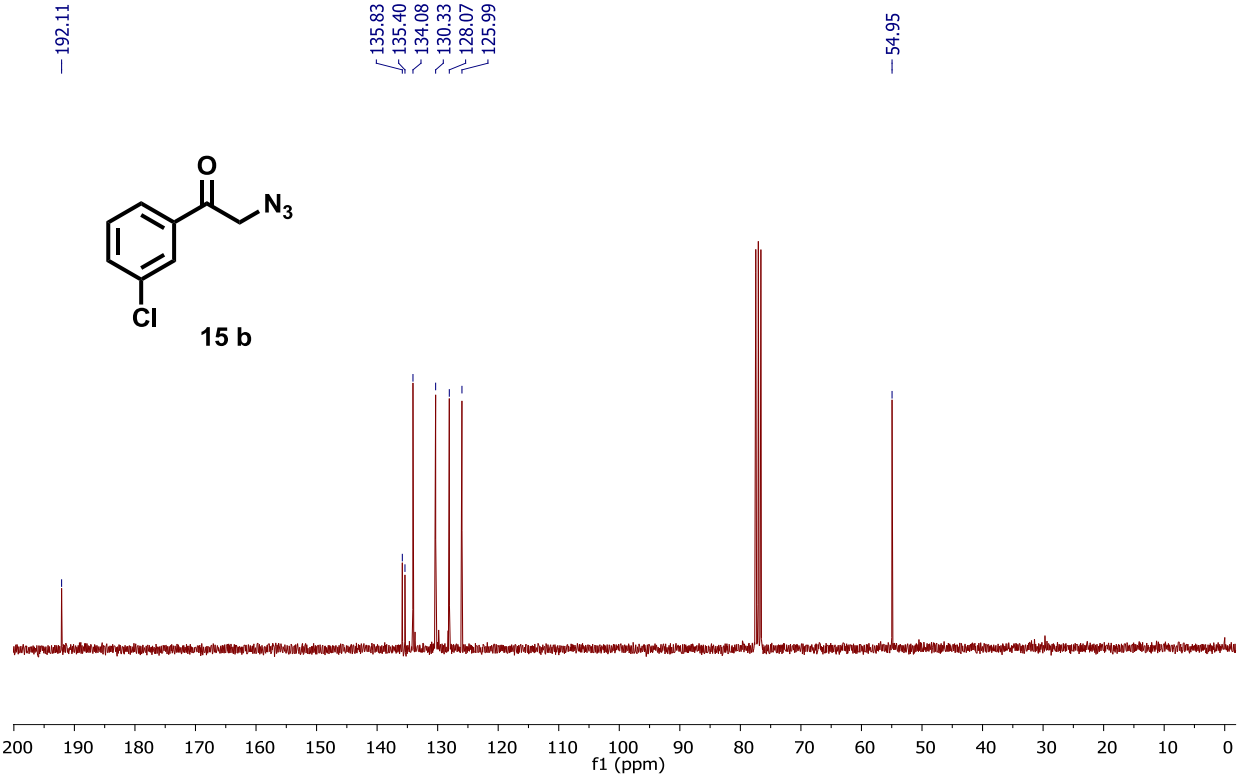
2-Azido-1-(4-chlorophenyl)ethanone



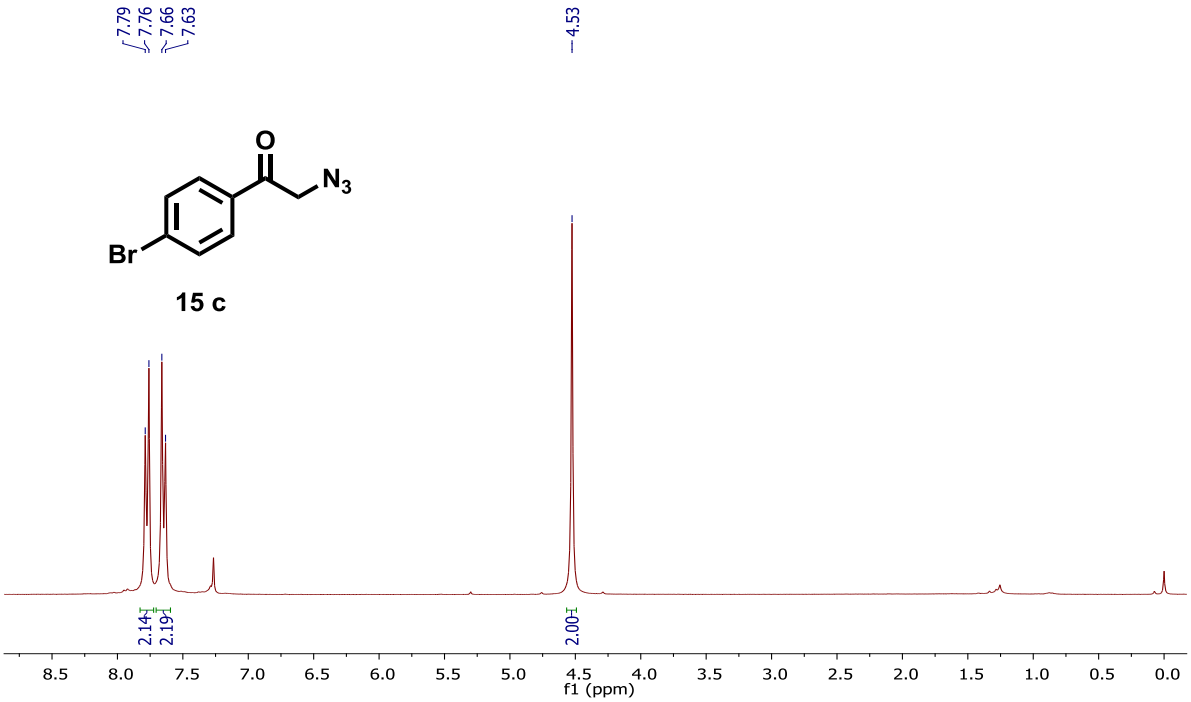



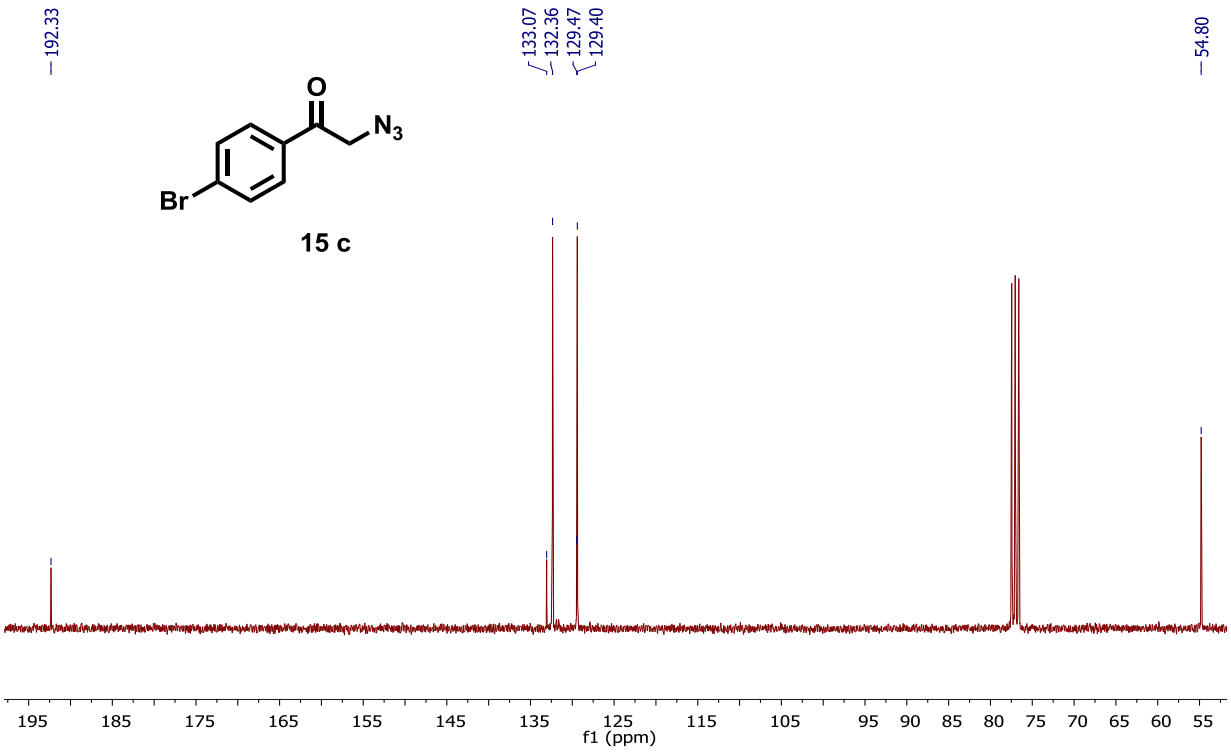
2-Azido-1-(3-chlorophenyl)ethanone



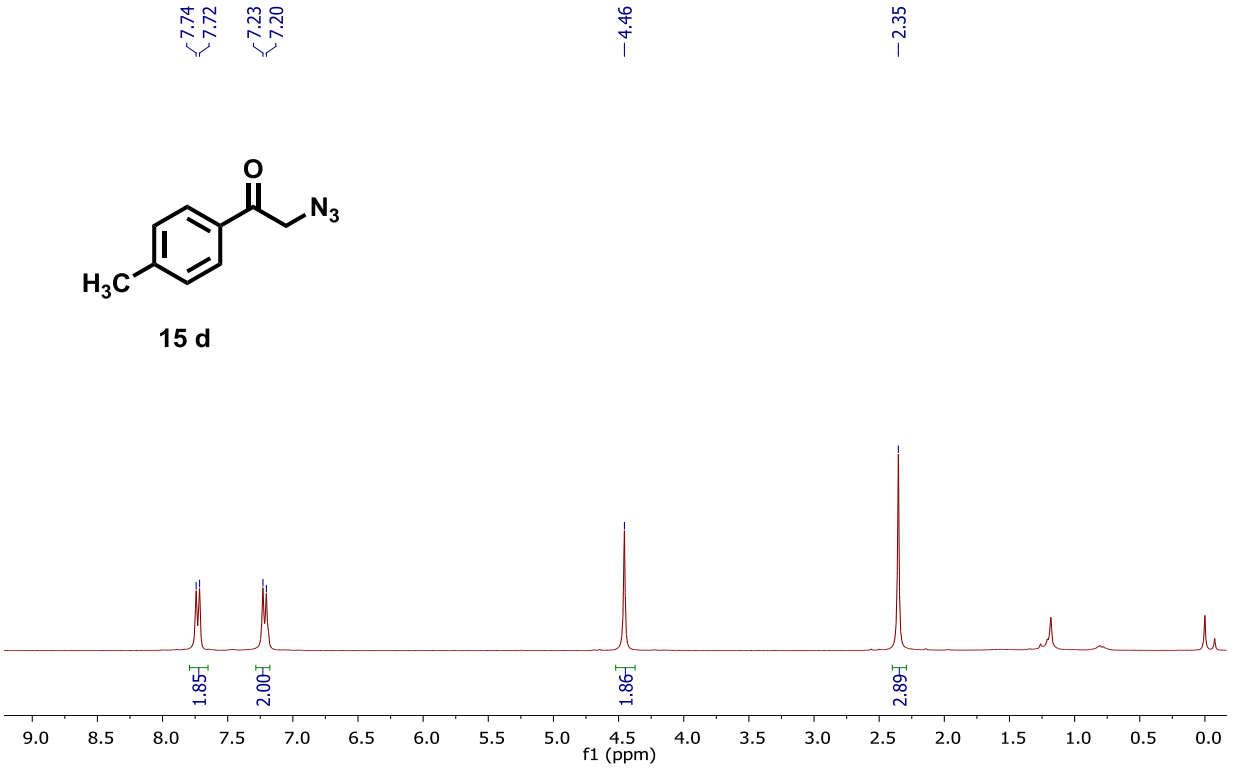



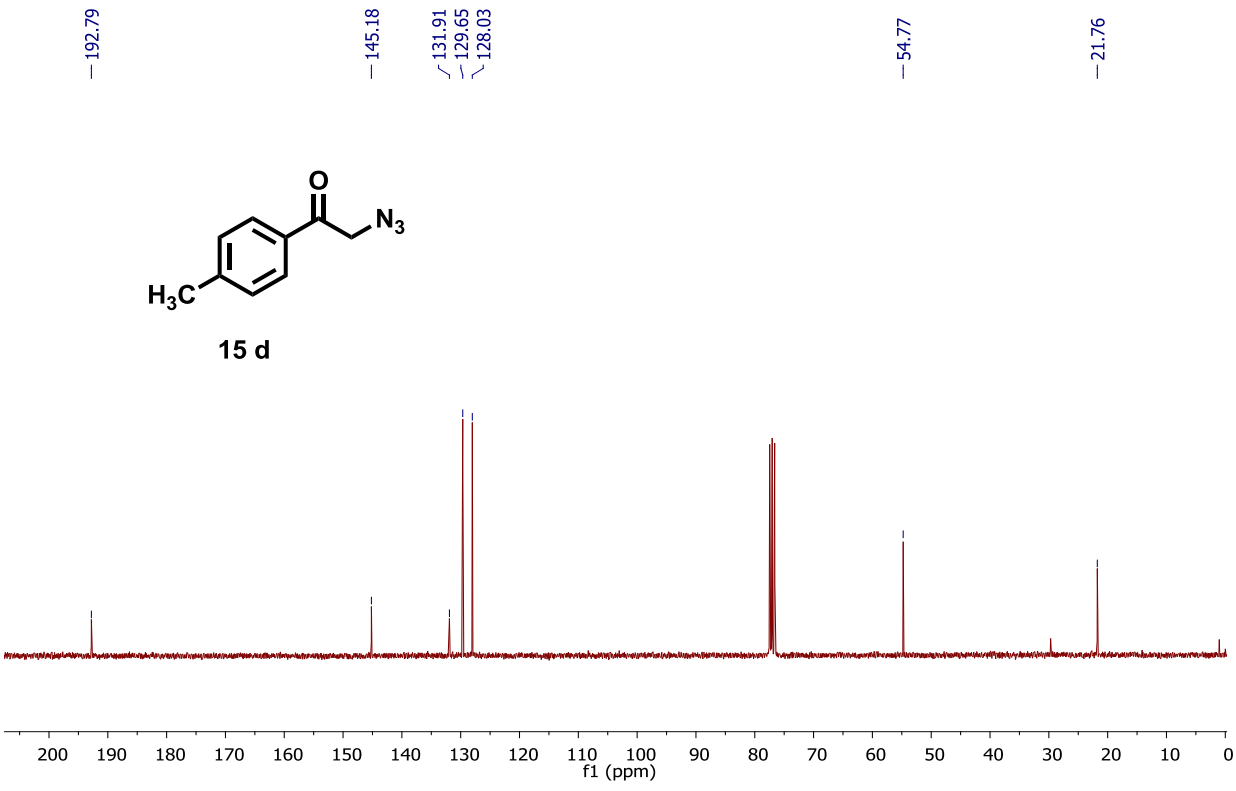
2-Azido-1-(4-bromophenyl)ethanone



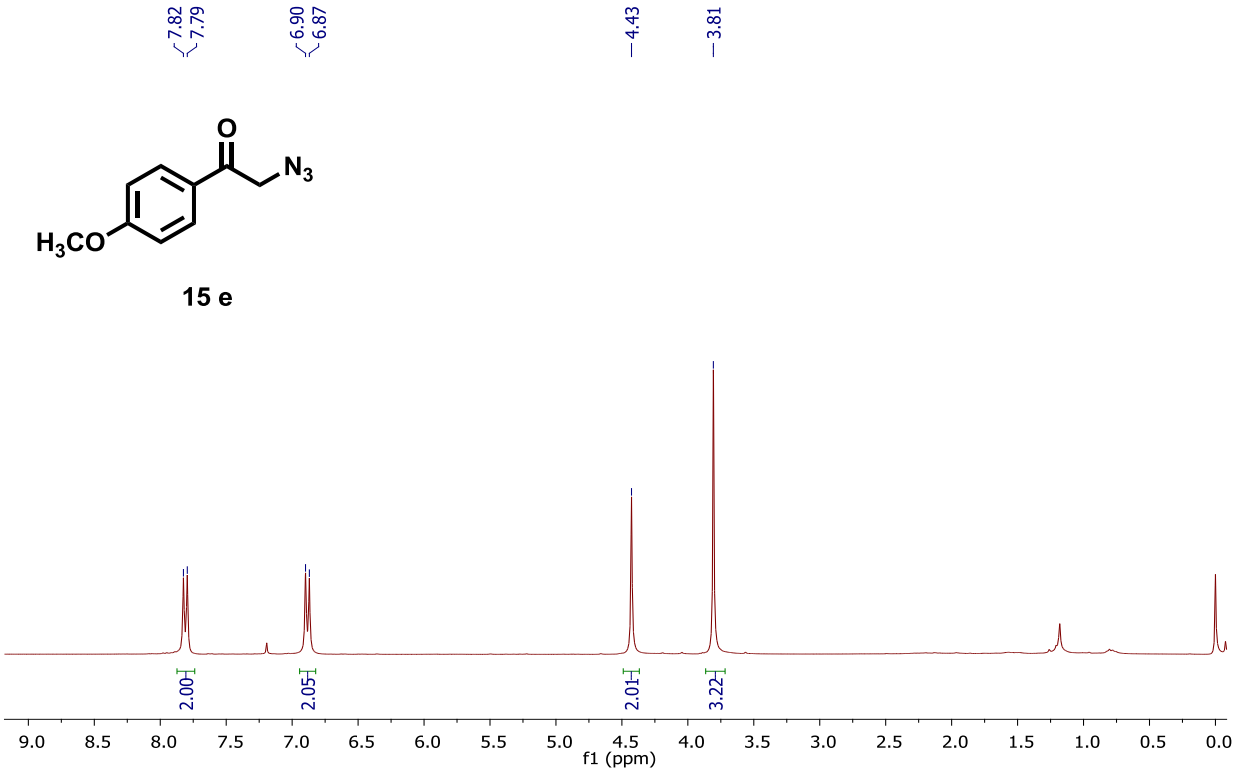



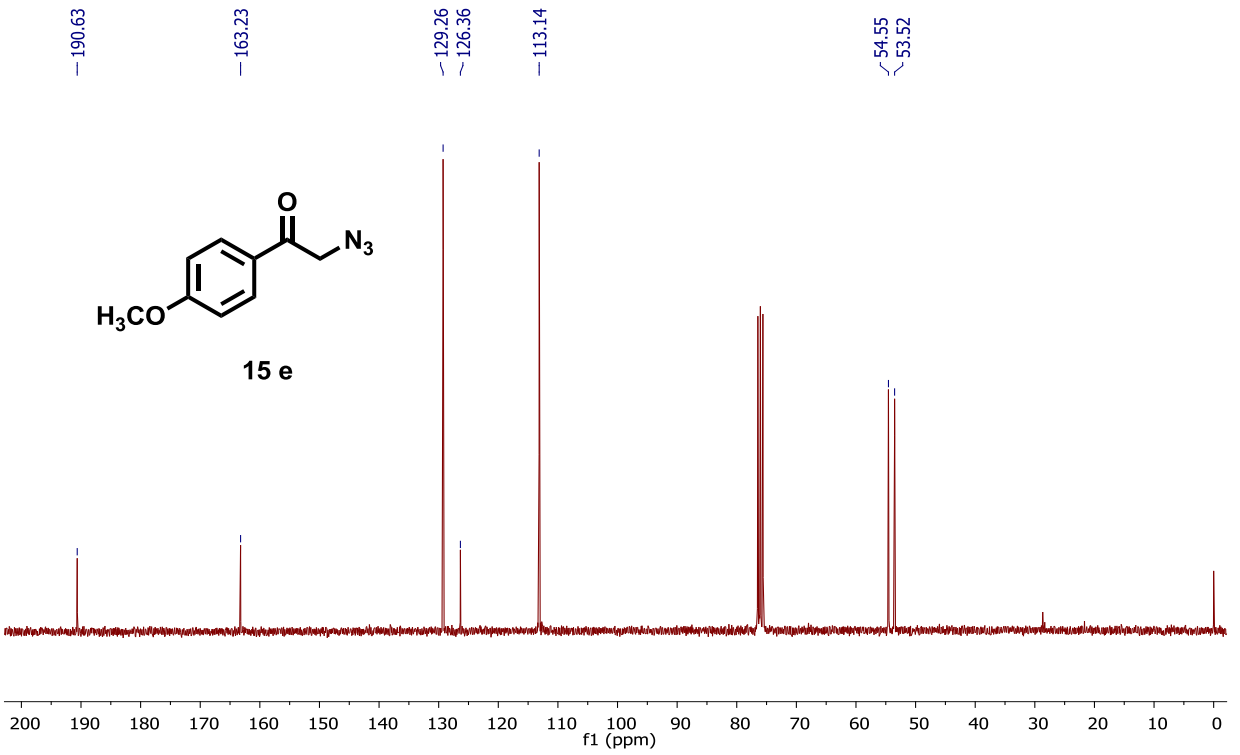
2-Azido-1-*p*-tolyl ethanone



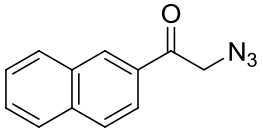



2-Azido-1-(4-methoxyphenyl)ethanone

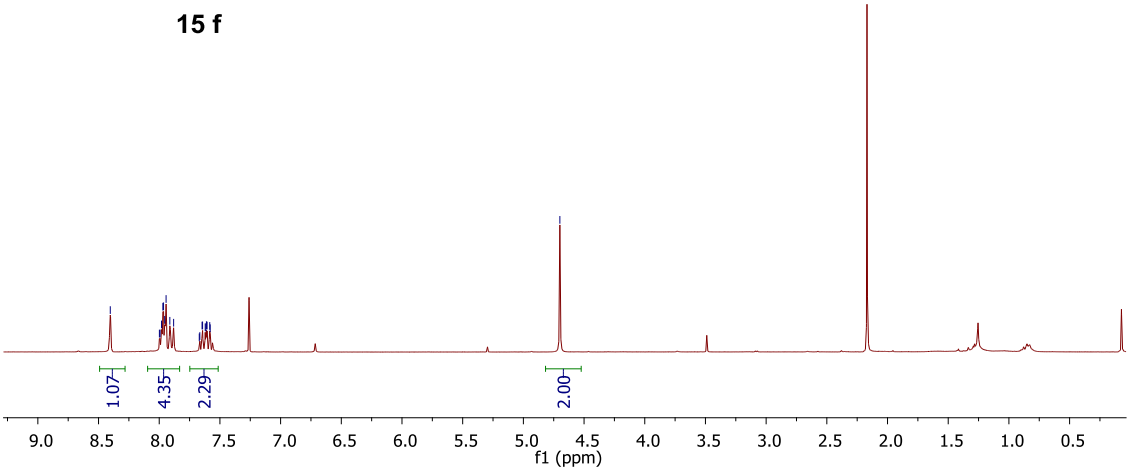


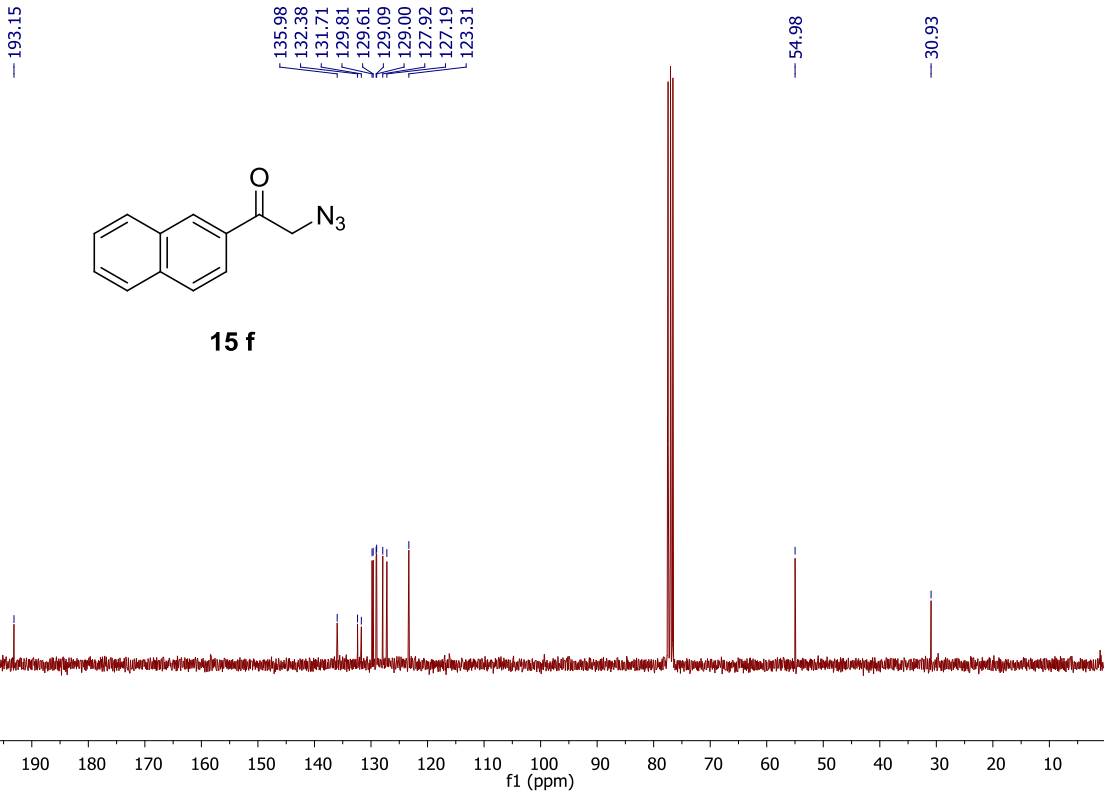



2-Azido-1-(naphthalene-2-yl)ethanone

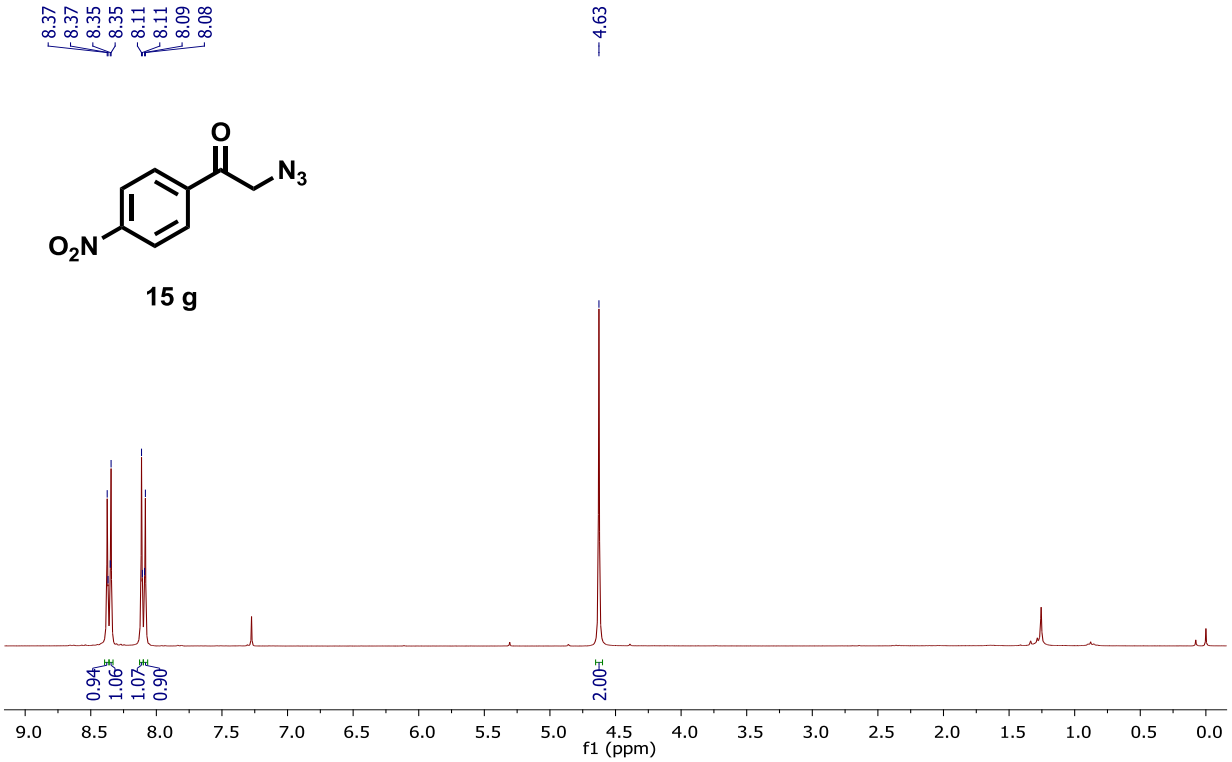



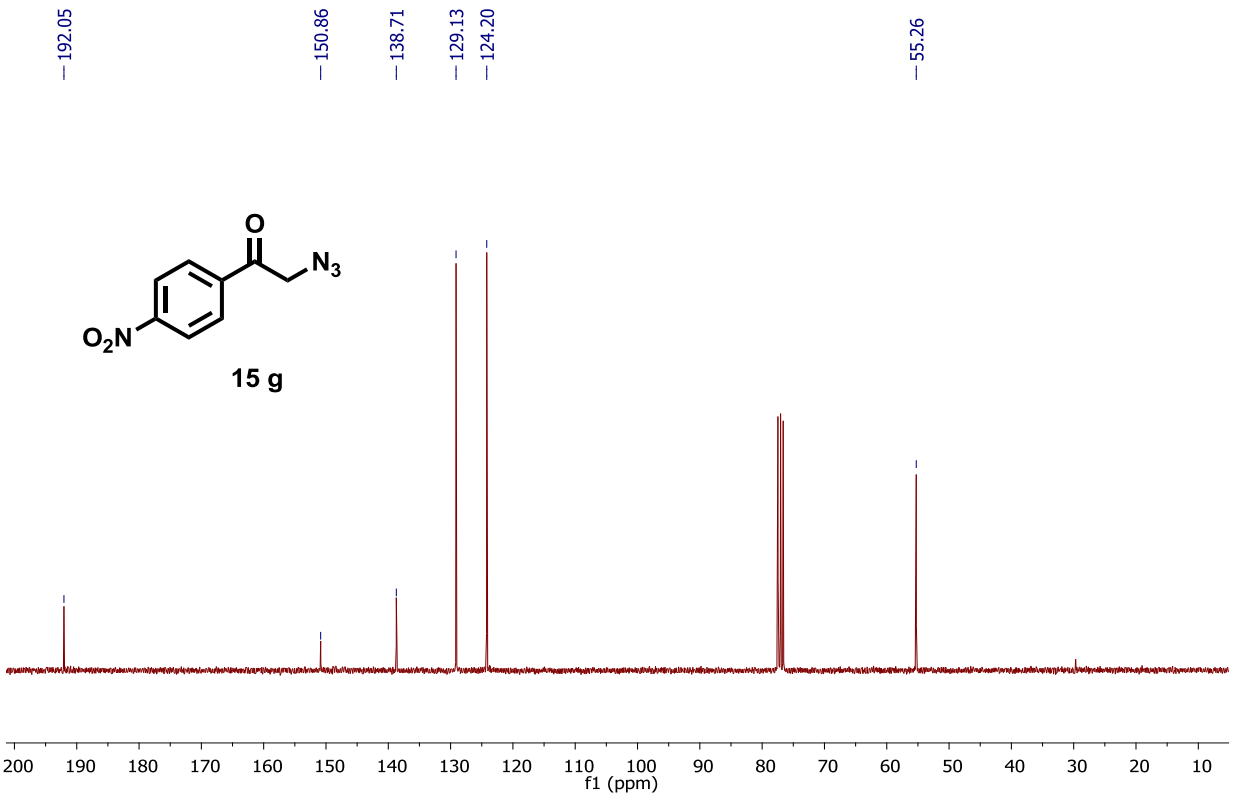

**15 f**



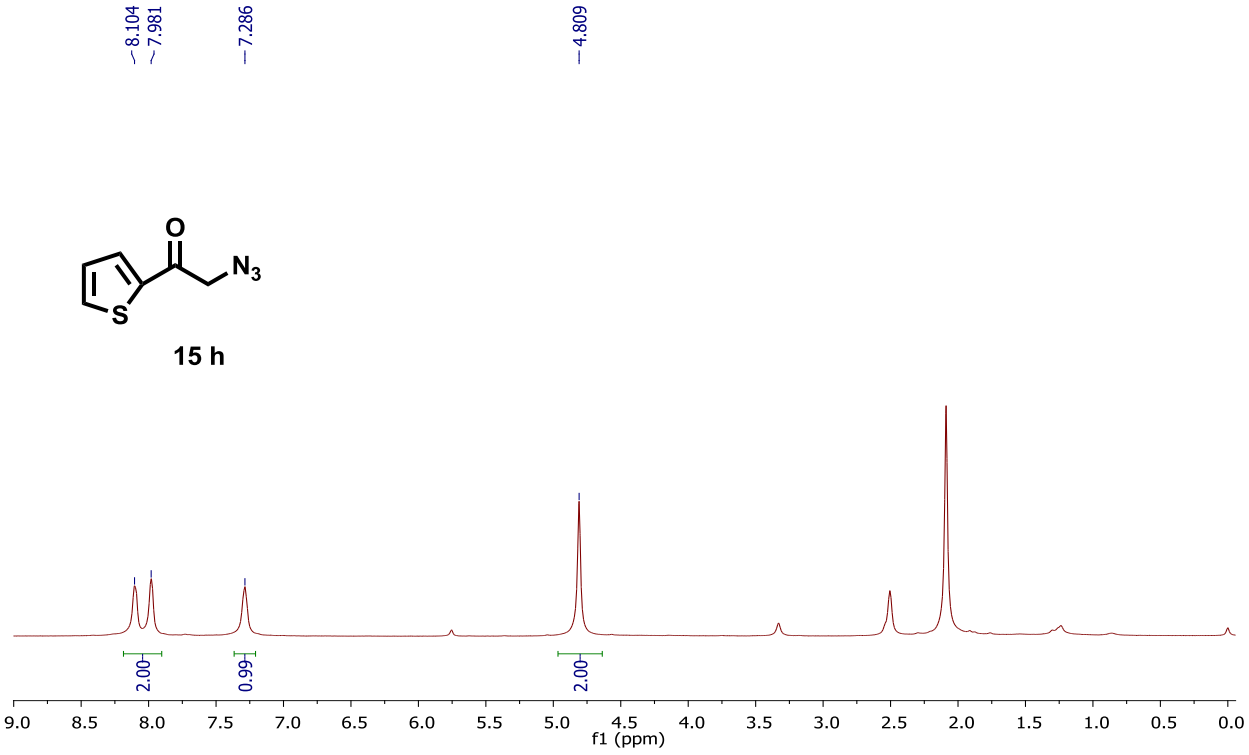



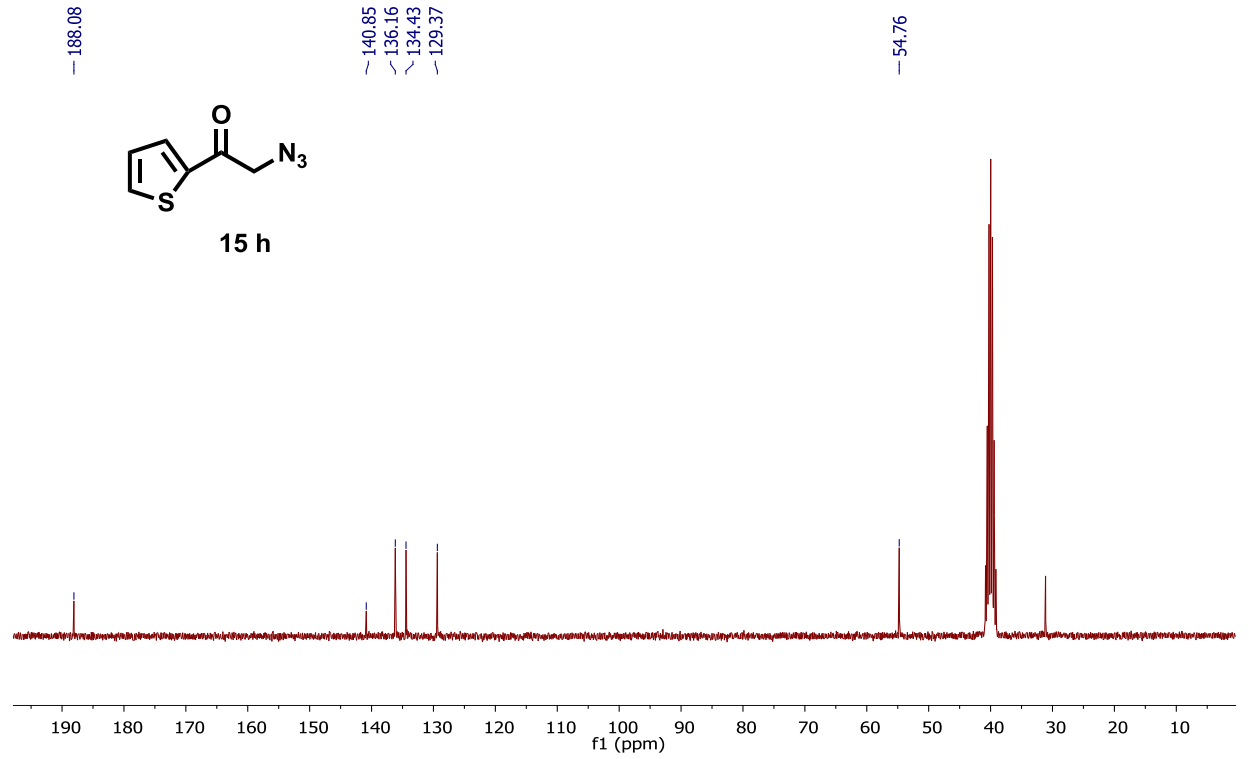
2-Azido-1-(4-nitrophenyl)ethanone



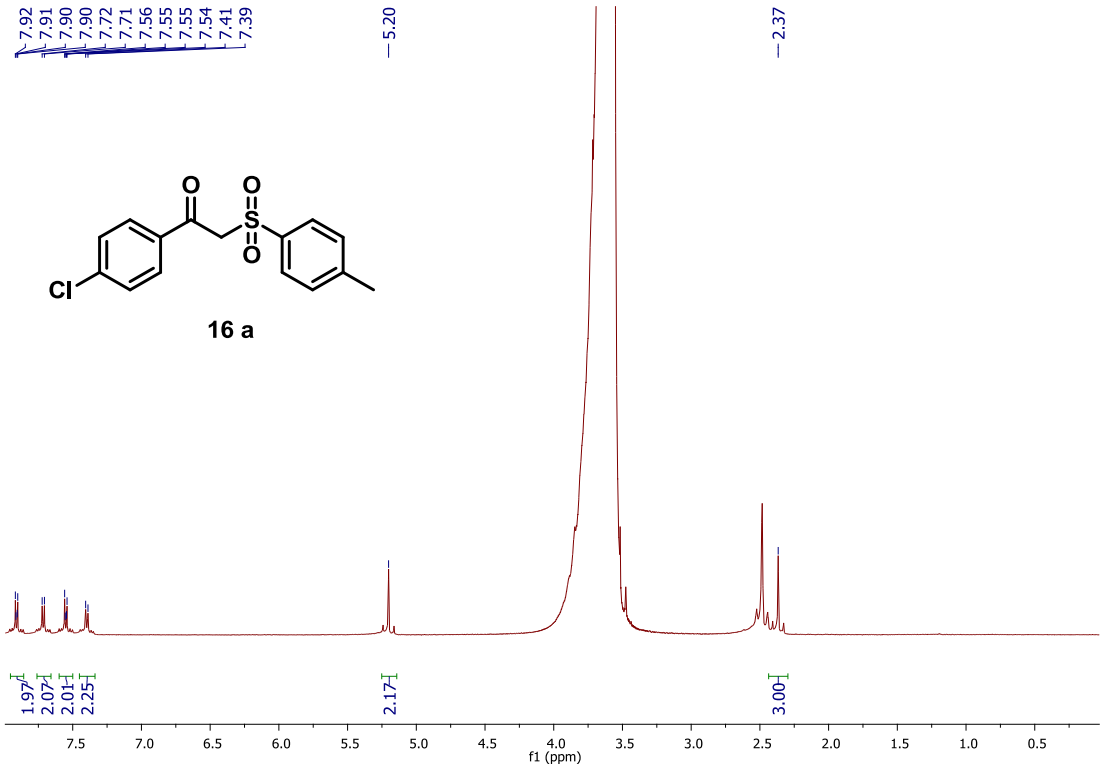



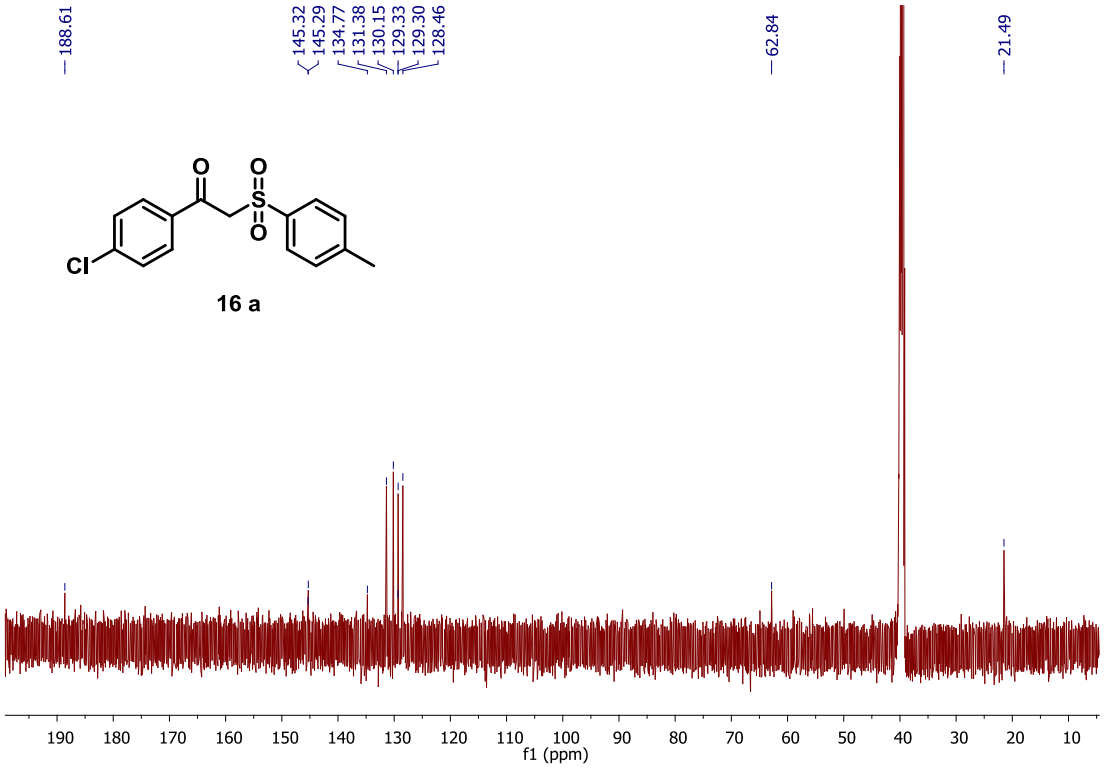
2-Azido-1-(thiophen-2-yl)ethanone



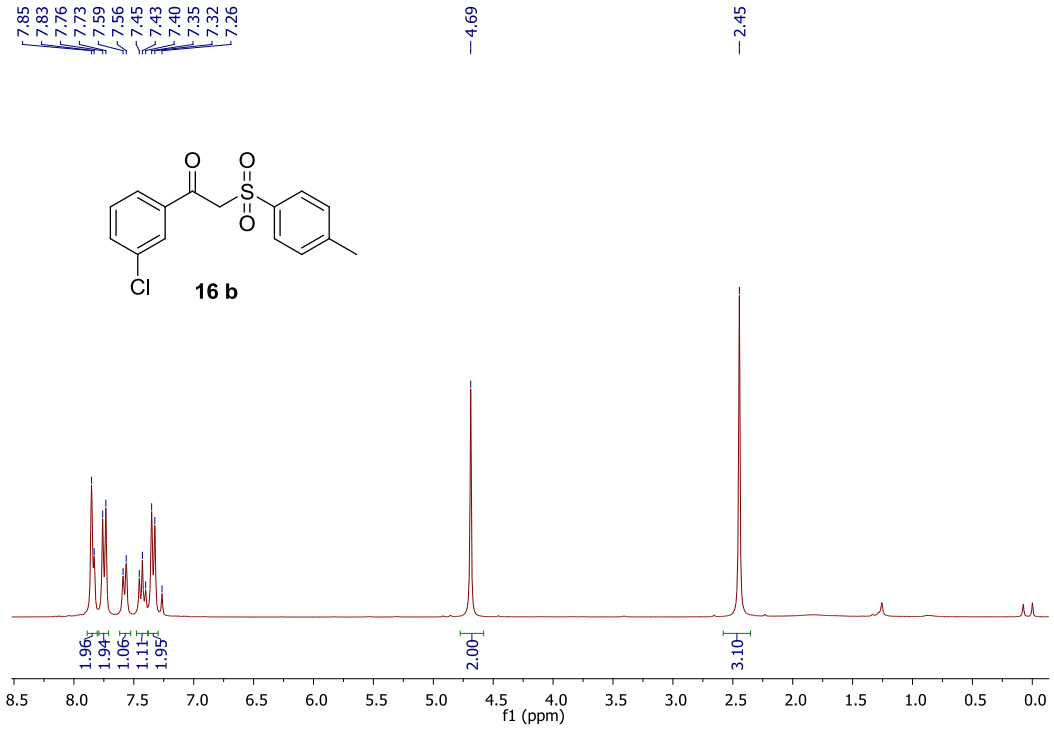



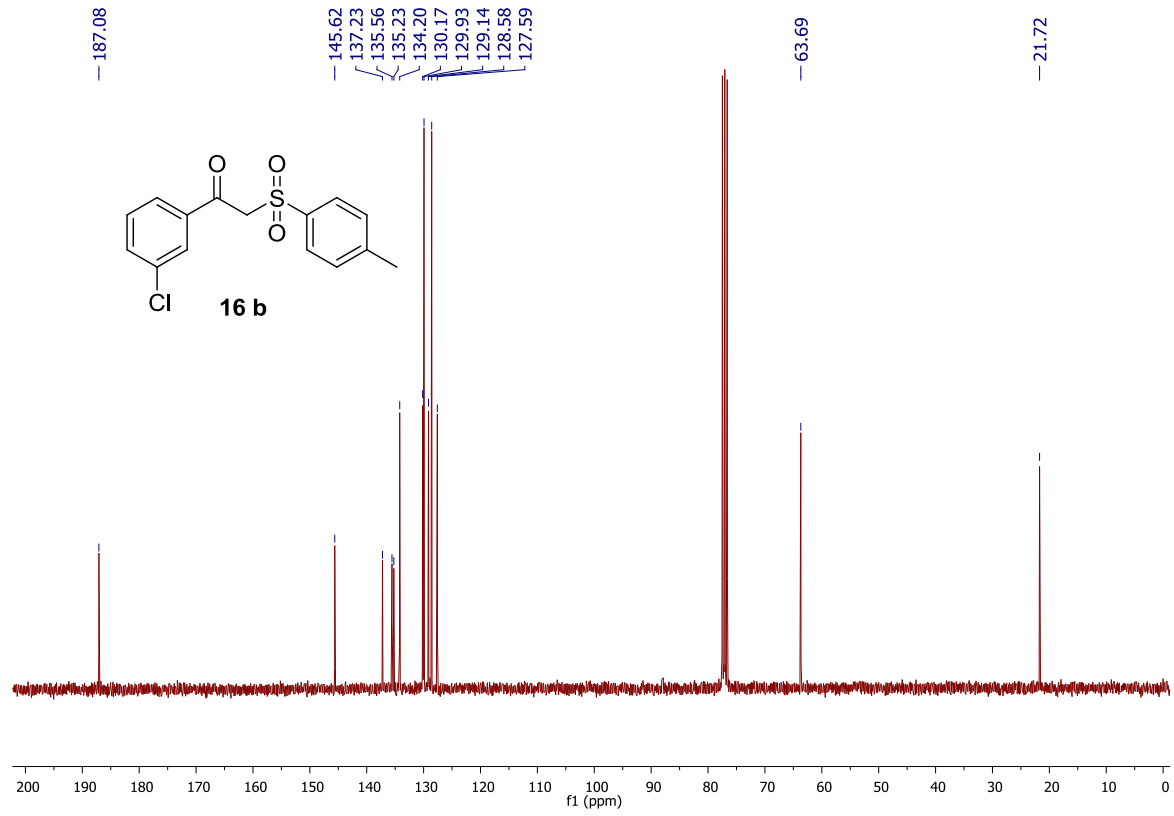
1-(4-Chlorophenyl)-2-tosylethanone



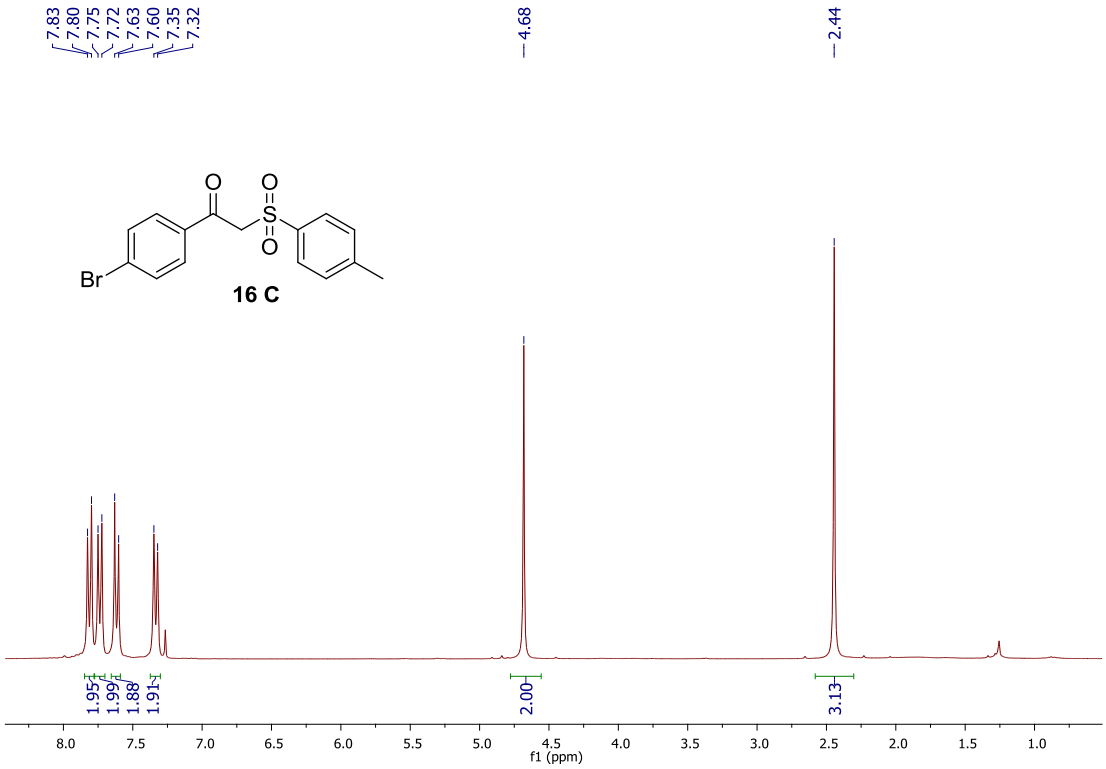



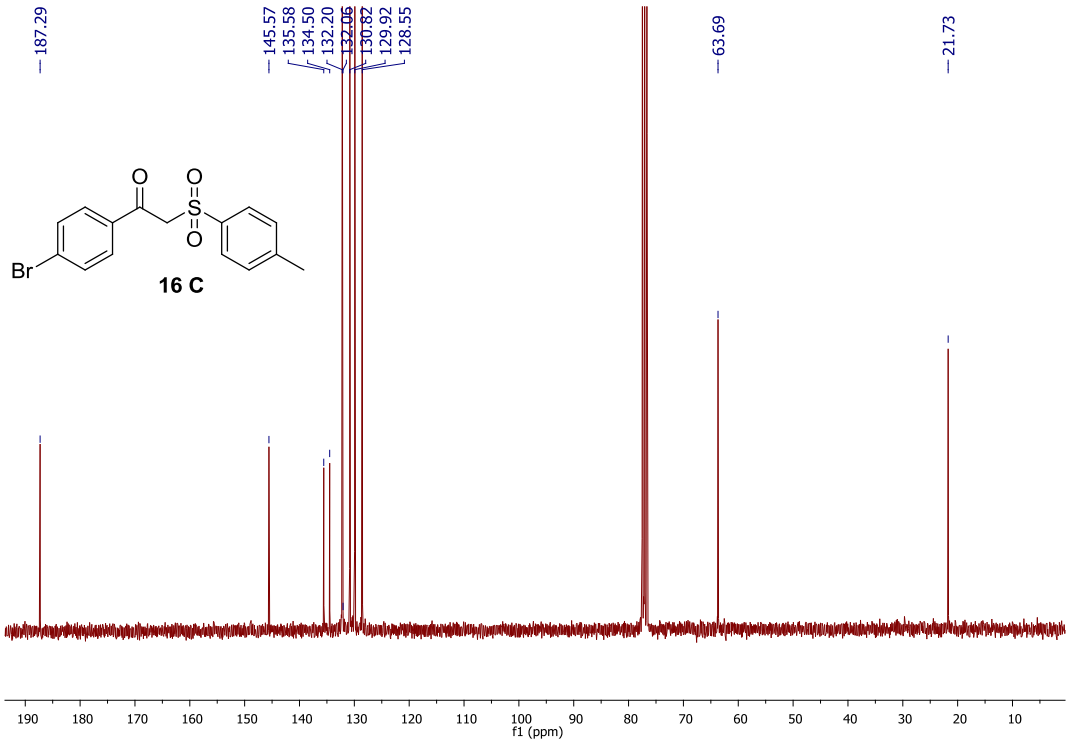
1-(4-Chlorophenyl)-2-tosylethanone



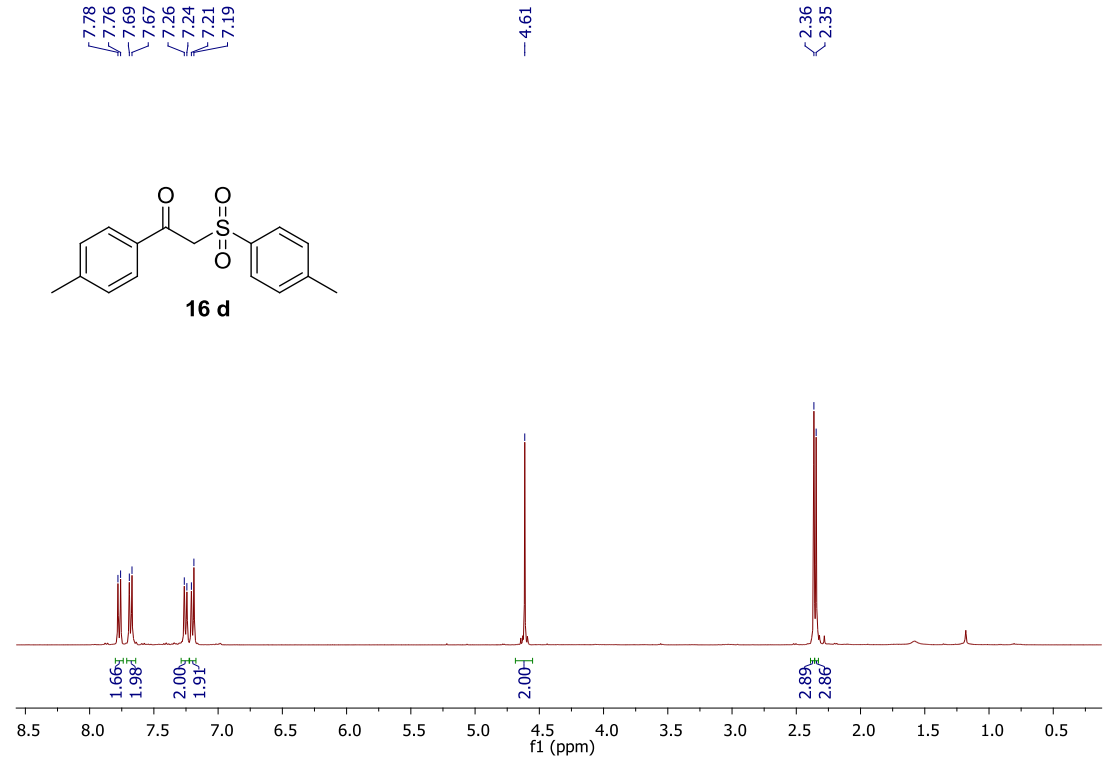



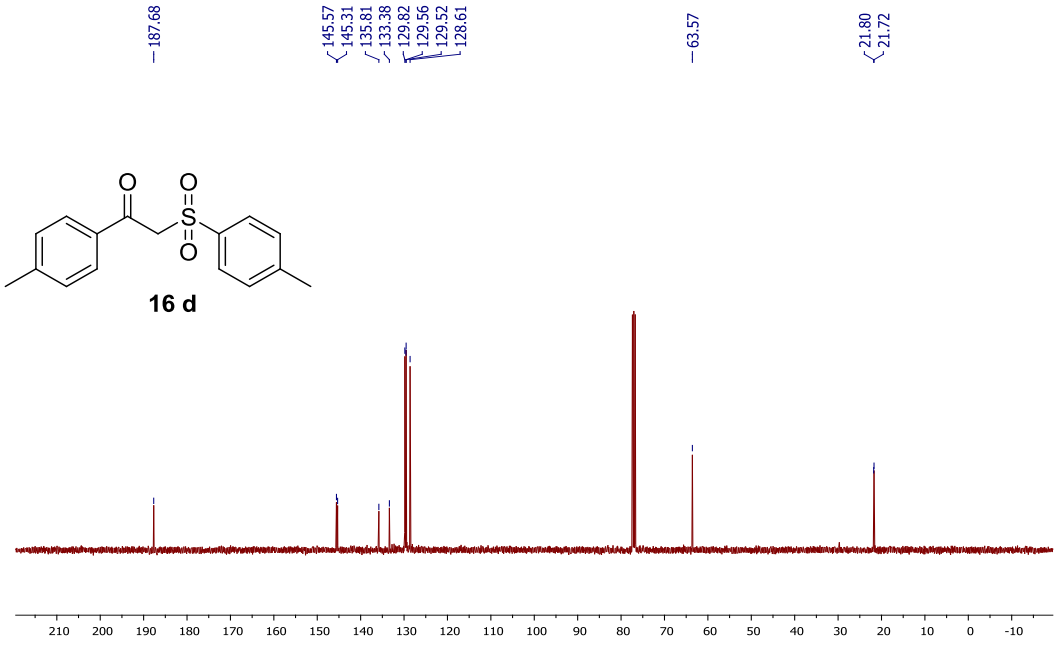
1-(4-Bromoorophenyl)-2-tosylethanone



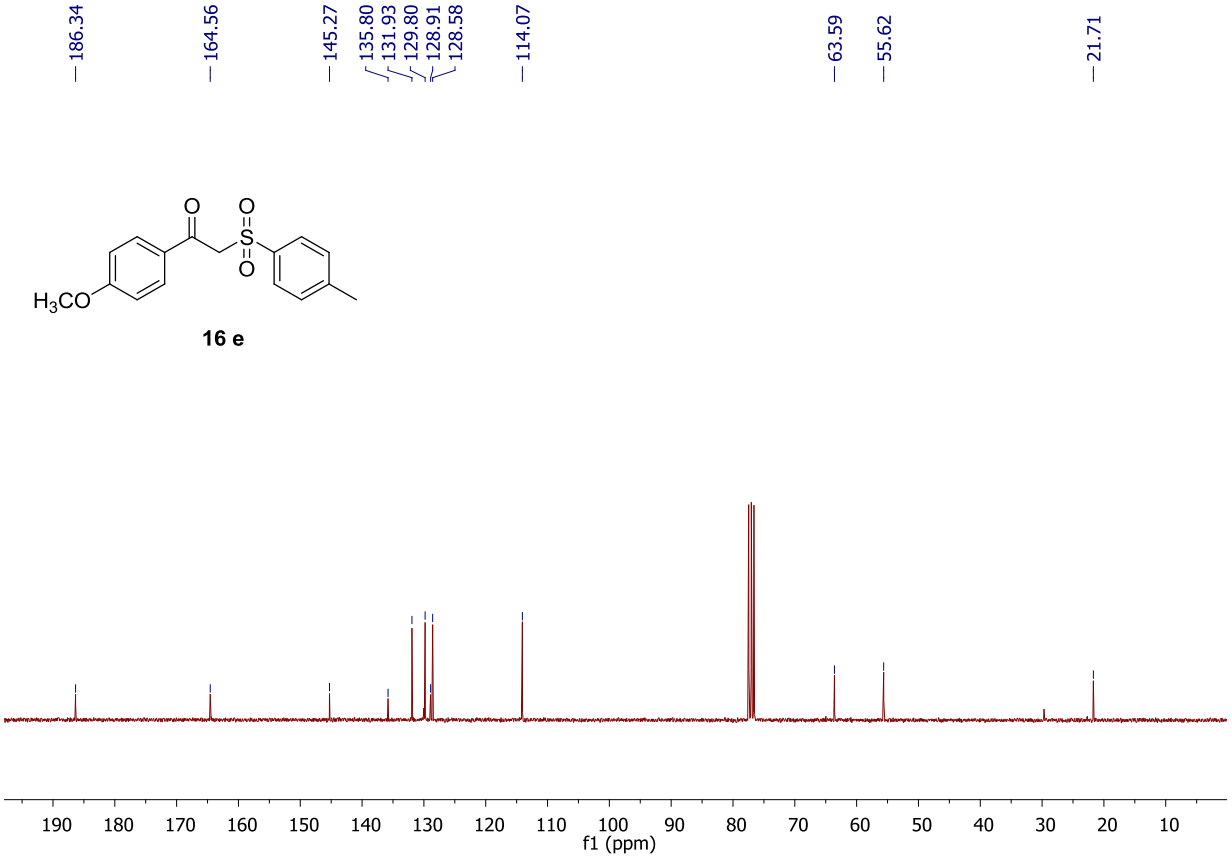



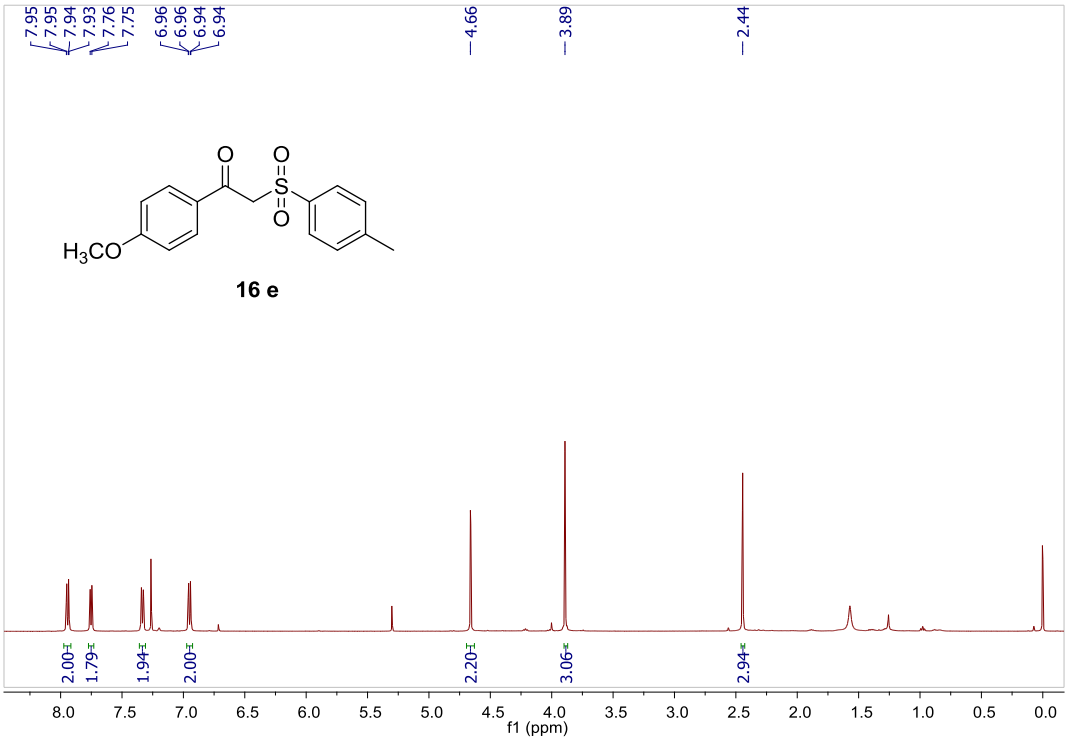
1-*p*-Tolyl-2-tosylethanone



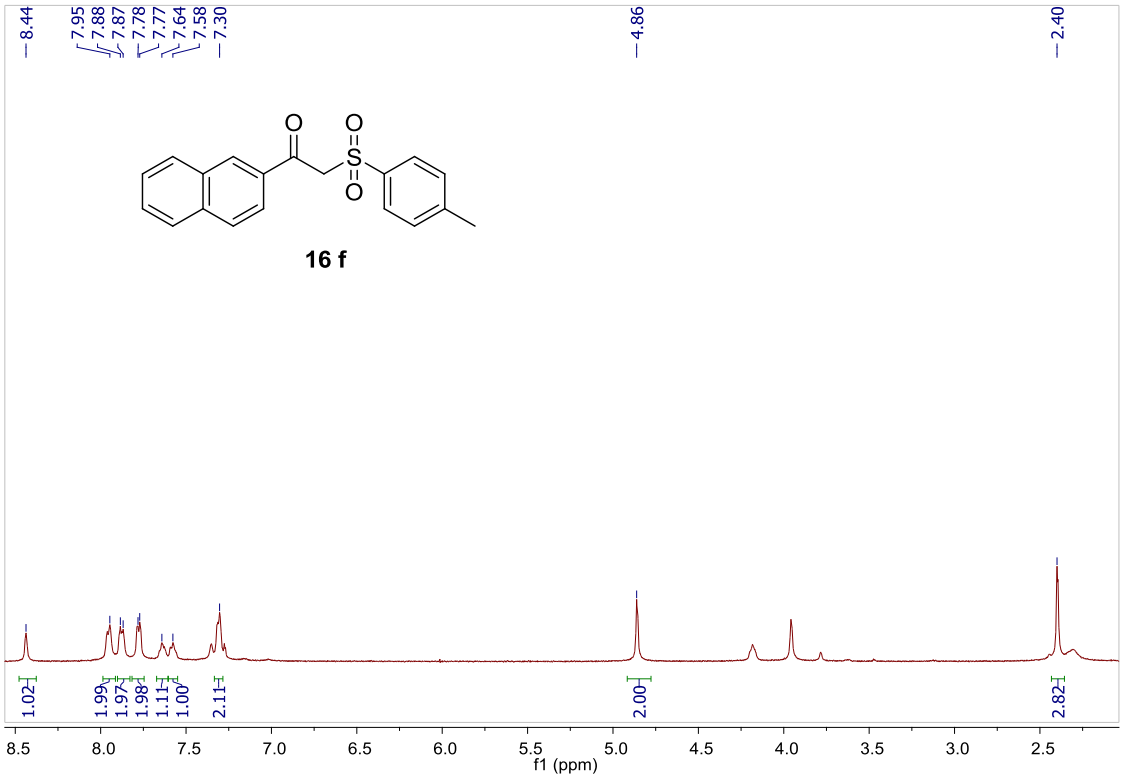



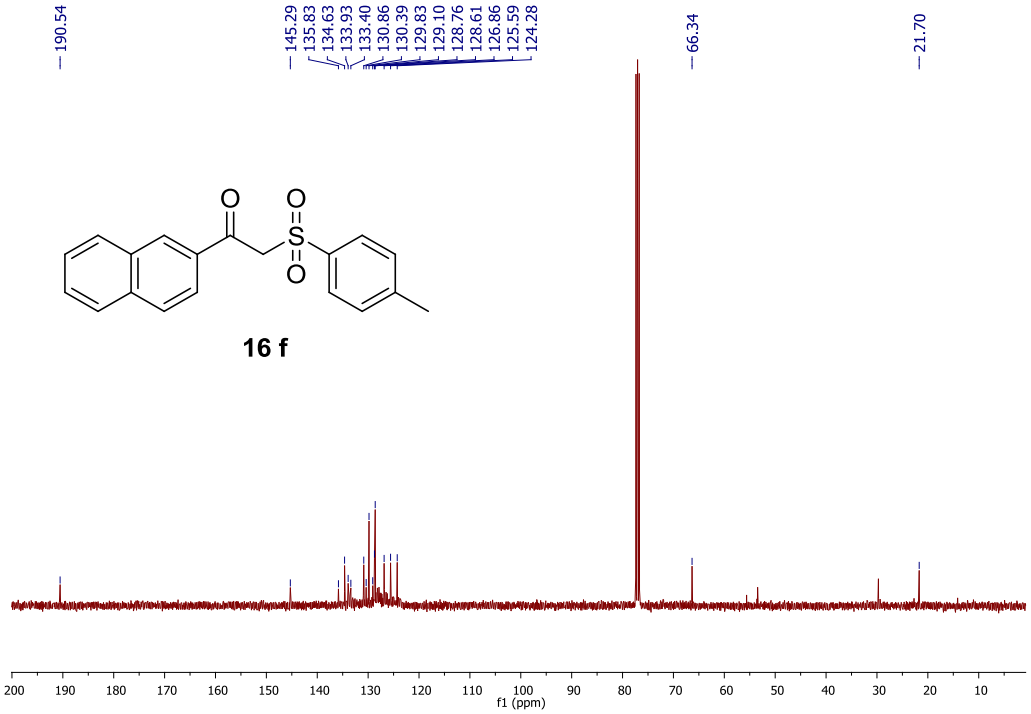
1-(4-Methoxyphenyl)-2-tosylethanone



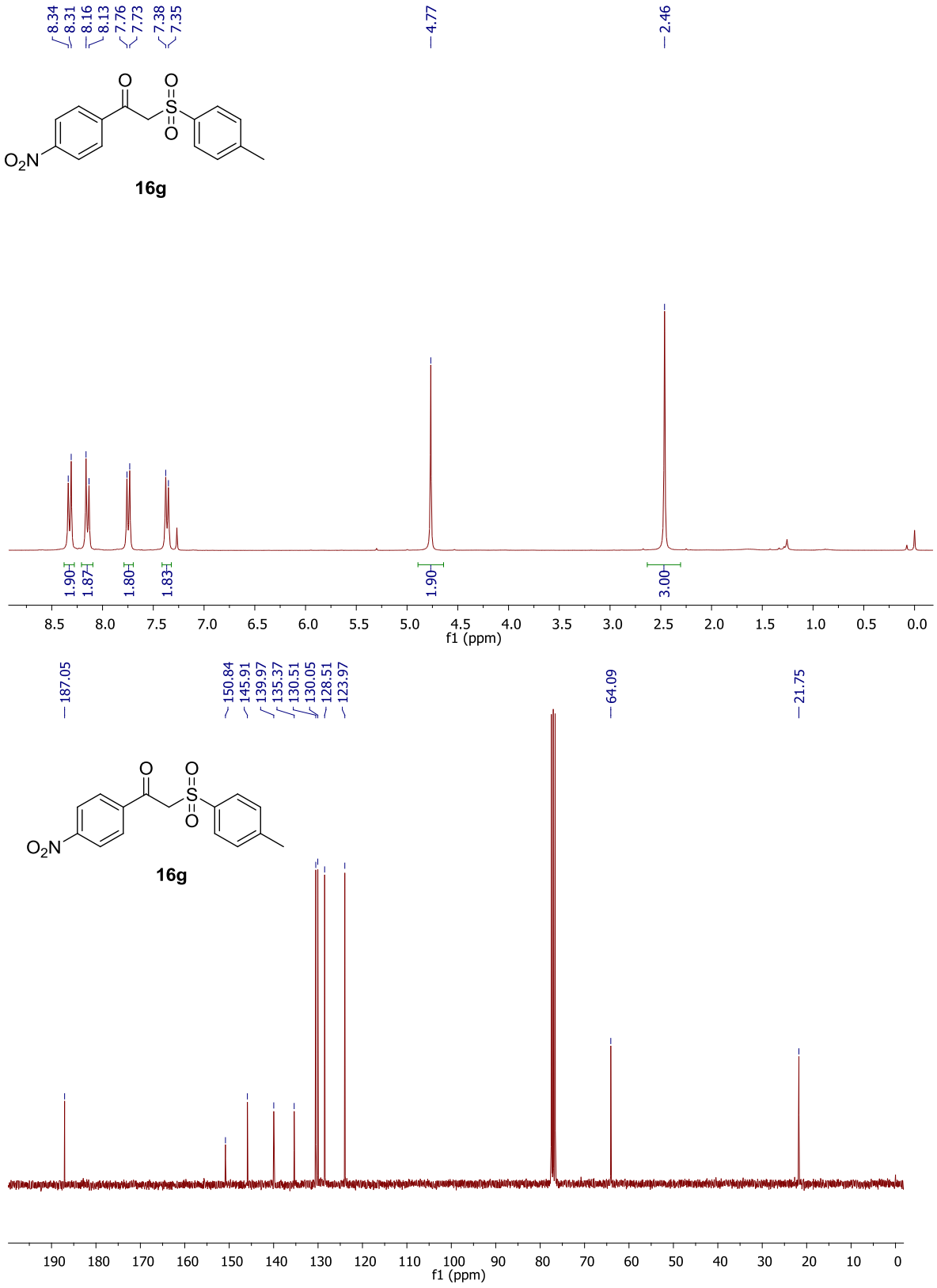



1-(Napthene2-yl)-2-tosylethanone

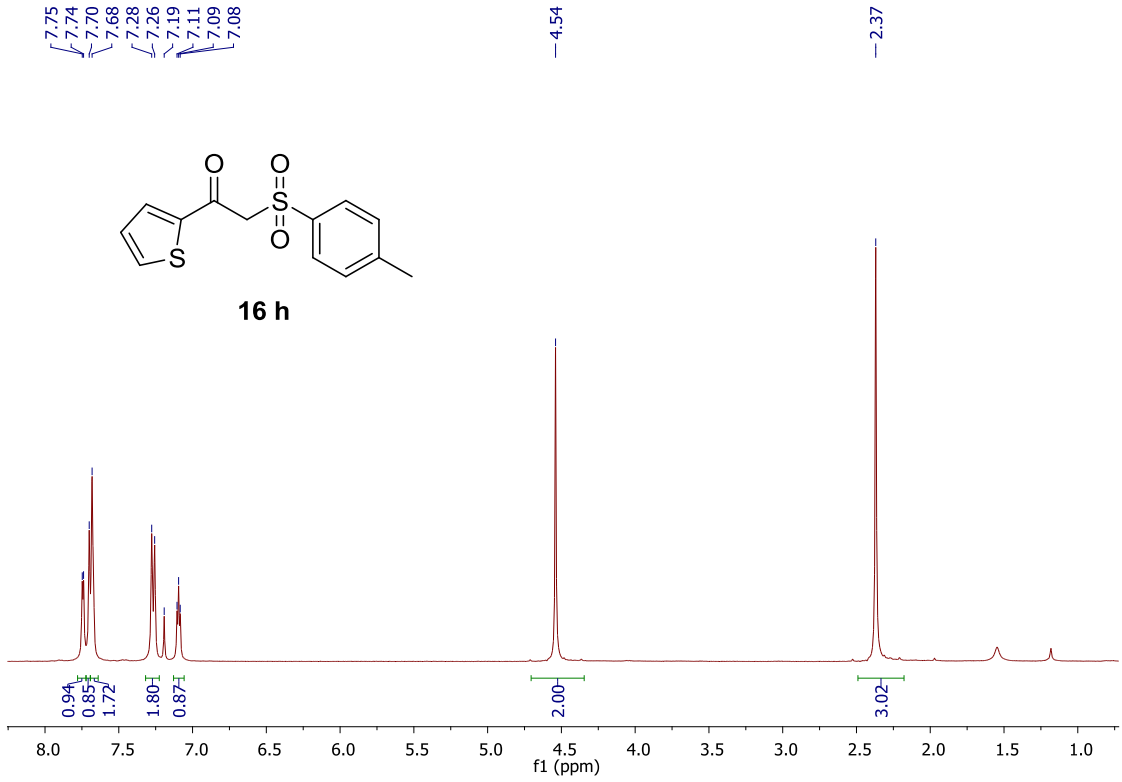


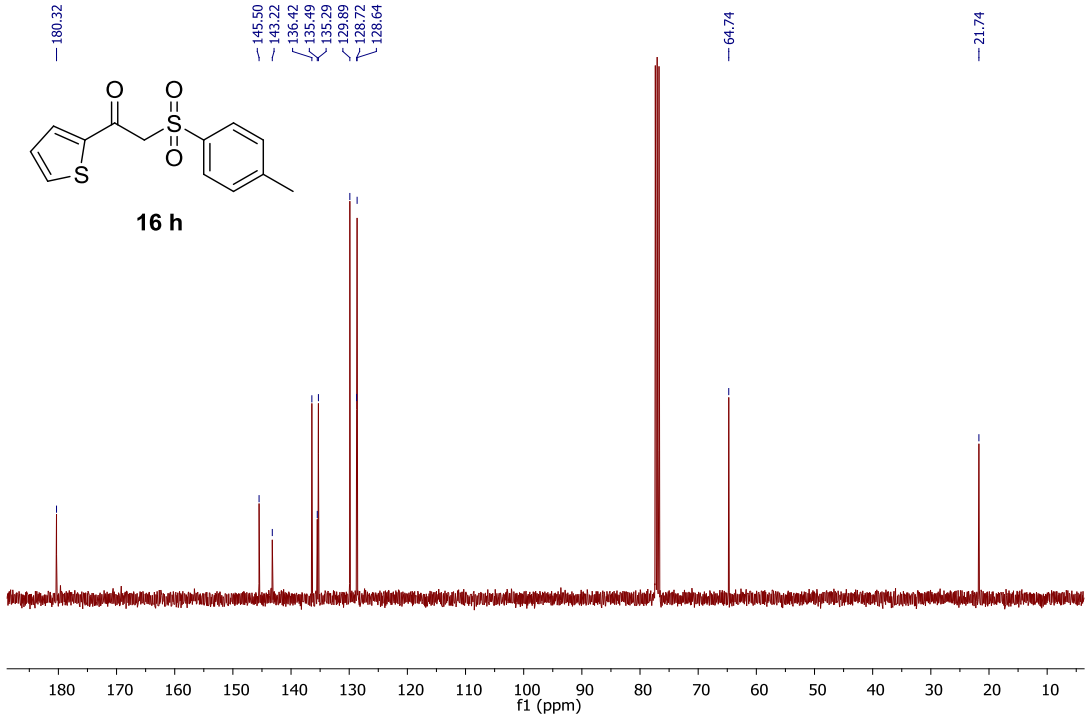



1-(4-Nitrophenyl)-2-tosylethanone



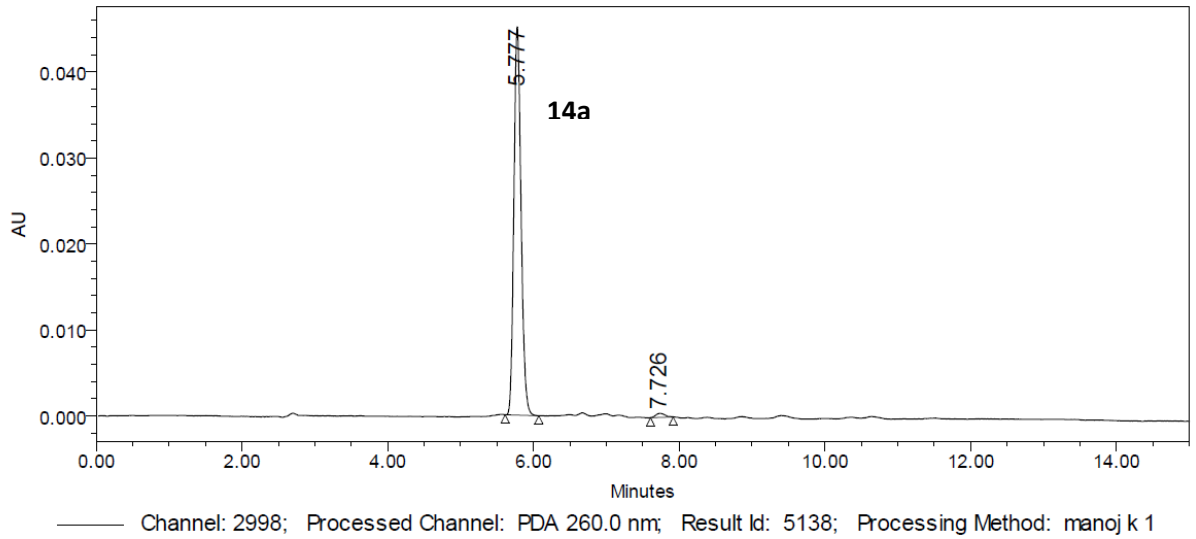
1-(Thiophen-2-yl)-2-tosylethanone



**3. HPLC analysis of 14a after extracting from reaction mixture and washing with water.**

| SAMPLE INFORMATION | | | |
|---|---|---|---|
| Sample Name: | KSCN | Acquired By: | System |
| Sample Type: | Unknow n | Sample Set Name: | |
| Vial: | 1 | Acq. Method Set: | mukund 1 half rate |
| Injection #: | 5 | Processing Method: | manoj k 1 |
| Injection Volume: | 10.00 ul | Channel Name: | 260.0nm |
| Run Time: | 15.0 Minutes | Proc. Chnl. Descr.: | PDA 260.0 nm |
| Date Acquired: | 9/1/2013 7:37:38 PM IST | | |
| Date Processed: | 9/1/2013 8:00:54 PM IST | | |




Channel: 2998; Processed Channel: PDA 260.0 nm; Result Id: 5138; Processing Method: manoj k 1

**Processed Channel Descr.: PDA 260.0 nm**

| | Processed Channel Descr. | RT | Area | % Area | Height |
|---|---|---|---|---|---|
| 1 | PDA 260.0 nm | 5.777 | 309228 | 98.71 | 45190 |
| 2 | PDA 260.0 nm | 7.726 | 4056 | 1.29 | 464 |